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Editor: Professor Kit Fai Pun
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**Note:** APETT’s logo was designed by Derek Aleong.
2 Editorial

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Editorial

This Issue (Volume 48 Number 1) of the Journal includes eight (8) articles. The relevance and usefulness of these articles are summarised below.

A.E. Faola et al., “The Use of Yam Stem Fibres as Bio-based Fillers in Polypropylene Composites for Structural Applications”, investigated polypropylene composites reinforced with yam stem particulate. The processed particles were used for the development of the polypropylene based composites using randomly dispersed open mould technique. Thermogravimetric analysis revealed that the treated yam stem particulate was more thermally stable in comparison with the untreated yam stem particulate while X-ray diffractograms analysis showed that alkaline treated peaks were more intense than the untreated. Scanning Electron Microscopy (SEM) analysis indicates improved adhesion in the morphology of the composites produced as there was no evidence of large agglomerate, delamination between the particles and the polymer matrix. Results obtained from the mechanical tests showed that the mechanical properties of polypropylene composites developed improved better than the unreinforced composites while the treated performed better than than the untreated composites.

In their paper, “A Study on Mechanical Properties of Aluminum-Graphite and Aluminum-Graphite-Zircon Hybrid Composites”, O.O. Taiwo et al., investigated the mechanical behaviour of Aluminum-based Graphite (Al-Gr) and the Al – Graphite – Zircon (Al-Gr-ZrSiO₄) composites under compressive loading. Using reinforcement of 4 and 8 wt.% each in Al-Gr and Al-Gr-ZrSiO₄ composites samples produced by stir casting and subjected to standard heat treatment, compression strength, modulus of compression, compression strain to failure, impact strength and micro hardness including density were evaluated. The results obtained show reduction in impact strength and compressive modulus with increased weight fraction of reinforcement, while other mechanical properties improve significantly with increase in wt. % fraction of reinforcement. For the Al-Gr composites, all the mechanical properties tested decreases with increase in weight fraction of the graphite reinforcement with exception of hardness which increase marginally.

O. Gbenenbor et al., “Strength and Wear Characteristics of Aluminum / Zircon / Graphite Composites”, investigated into the failure of monolithic alloys with a need for stable structural integrity of materials. In this study, varying amount of zircon, graphite and the combination of the two (hybrid) particles were used as reinforcement in aluminium 6011 alloy. The cast samples were characterised for compressive strength, hardness, wear and morphological responses. The morphology showed fine crystals of AlFeSi with increase in the concentration precipitated Mg₂Si phase in the aluminium matrix. Fluctuations in the wear characteristics of composites were observed. It was claimed that the composites could serve as potential materials for light automobile brake pads.

T.T. Areo et al., “Thermal Performance of Different Shapes of Solar-powered Oven for Meat Processing and Preservation”, investigated heat energy generation through the radiation of solar energy in a confined space. In this study, the thermal performance of different geometries of a solar oven and the drying of meat samples in the device were analysed. The curve-type box model was adopted and used in fabrication for experimental analysis. The numerical results were compared to the results from the experiment conducted. The outcome of this comparison showed that the curve-type solar oven design with the heat samples largely spaced ensured the drying effect. The meat samples for the optimal design dried uniformly after 40 minutes and the average percentage difference between the numerical and experimental results was approximately 2.75%. The findings would shed lights in improving the effectiveness and processing time of existing box solar ovens.

S.O. Ogunrinde, O. Babatunde, and D.S. Aribike, “Study of Effects of Afraegle Paniculata Extract on Corrosion of Galvanised Steel in H₂SO₄ Solution”, investigated the reaction mechanisms of the extract and explored the effects of the kinetics and thermodynamics of adsorption step in the corrosion reaction. Using weight-loss and gasometric techniques, scanning electron microscope (SEM) and energy dispersive X-ray spectroscopy (EDS), the acidic medium increased the corrosion rate of galvanized steel and the corrosion reaction. The adsorption of the extract on galvanised steel surface obeyed the Langmuir adsorption isotherm with both reaction rate and Standard Change in Gibbs Free Energy of adsorption increasing with increase in temperature. The reaction was thermodynamically feasible and spontaneous. The SEM images also confirmed that galvanised steel corroded in acidic environment and the extract inhibited the rate of corrosion in galvanised steel in H₂SO₄ solution. The findings suggested that corrosion rate could be reduced by using A. Paniculata extract as an inhibitor.

In their paper, “Effects of Micro Silica and Waste Glass on the Rheological and Mechanical Properties of Self-Compacting Concrete”, T.A. Buari, F.A. Olutoge and C. Egwanwor, studied the properties of the concrete. The rheological characteristics of the concrete were evaluated while the compressive strength was determined at 7, 14, 21, 28 and 56 days curing by crushing. Followed the setting of experimental procedures and the EFNARC (2002) Specification and Guidelines for Self-Compacting Concrete, it was found that the slump flow decreased with
increased inclusion of MS, T50cm and V-funnel flow time increased with increased replacement, while L-box values decreased indicating reduction in passing ability, this is due to the high water absorption capacity of MS. The reverse was the case when waste glass (WG) was introduced into the concrete matrix due to the low water absorption capacity of WG compared to river sand as evident in water absorption test.

D. Ramkissoon and K.F. Pun, “Assessing Human Factors at the Design Stage of Upstream Oil and Gas Projects: A Risk Assessment Methodology”, explored the use of a standardised risk assessment approach to address human factors at the design stage in the oil and gas industry. It aimed to utilise the approach to develop a proposed methodology to assess human factors, thus making the engineering design safer. The methodology constitutes 9 steps to evaluate human factors in engineering design and to assess the risk posed by human error in the oil and gas sector. A case study of installing a pressure transmitter on a flowline of the oil and gas platforms was used, and an activity nonetheless which if performed incorrectly can result in a loss of containment of hydrocarbon gas. Utilising the proposed methodology, the activity’s constituent tasks are diagnosed and each task would now be subject to analysis by showing the consequences of human error.

P. Raeburn-Marcano and K.F. Pun, “Assessing Small Enterprises’ Maturity Levels of Knowledge, Attitudes and Practices towards Emergency Preparedness in Trinidad: A Pilot Study”, reported the findings of a pilot study that examined small enterprises’ knowledge, attitudes and practices (KAP) towards emergency preparedness in Trinidad and Tobago (T&T). It was found that lack of legislations, limited financial resources and little institutional guidance were amongst the main challenges for small enterprises in engaging emergency preparedness operations (EPO). A 5-level Generic Emergency Management Assessment (GEMA) approach was developed with implementation guide. The approach would serve as a guidance tool for small enterprises to 1) assess their current maturity level, 2) implement programme requisites to bridge gaps, and 3) move from one maturity level to the next.

Acknowledgements

On behalf of the Association, we gratefully acknowledge all authors who have made this issue possible with their research work. We greatly appreciate the voluntary contributions and unfailing support that our reviewers give to the Journal. Our reviewer panel is composed of academia, scientists, and practising engineers and professionals from industry as listed below:

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October 2020
The Use of Yam Stem Fibres as Bio-based Fillers in Polypropylene Composites for Structural Applications

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Abstract: This study investigates polypropylene composites reinforced with yam stem particulate. Yam stem was obtained from the stem of harvested white yam tuber (Dioscorea spp.) and was treated with sodium hydroxide (NaOH). Both treated and untreated yam stem particles were pulverised to obtain particle sizes of <150 µm. The constituents were analysed by gravimetric method while Thermogravimetric Analyser (TGA) and X-ray Diffractograms (XRD) were used for characterisation. The processed particles were used for the development of the polypropylene based composites using randomly dispersed open mould technique. The result showed that chemical treatment of the particles with NaOH enhanced the removal of lignin and hemicellulose and, the retention of the cellulose essential for reinforcement. TGA analysis revealed that the treated yam stem particulate was more thermally stable in comparison with the untreated yam stem particulate while XRD analysis showed that alkaline treated peaks were more intense than the untreated due to the chemical treatment which was able to remove the unwanted constituents from the particle surface thus, exposing the cellulose which is highly crystalline. Scanning Electron Microscopy (SEM) analysis indicates improved adhesion in the morphology of the composites produced as there was no evidence of large agglomerate, delamination between the particles and the polymer matrix as well as pull out of the particles from the matrix. Results obtained from the mechanical tests showed that the mechanical properties of polypropylene composites developed improved better than the unreinforced composites while the treated performed better than the untreated composites. Optimum mechanical properties were achieved at 20 wt.% particulate reinforcement of the polypropylene.

Keywords: Biodegradable material, eco-friendly, structural material, pollution control, environmental impact

1. Introduction

Polypropylene (PP) is a synthetic, high molecular mass linear addition polymer of propene. It is an economical material that offers a combination of outstanding physical, mechanical, thermal, and electrical properties not found in any other thermoplastic. Compared to low or high density polyethylene, it has a lower impact strength, but superior working temperature and tensile strength (Beckermann and Pickering, 2008). Noted for its excellent chemical resistance in corrosive environments, polypropylene provides excellent resistance to organic solvents, degreasing agents and electrolytic attack (Cao et al., 2006). It is light in weight, resistant to staining and has a low moisture absorption rate. It is one of the most commonly used polymers, and has good mechanical properties, heat resistance, low cost, ease of processing, and full recyclability. Polypropylene provides superior qualities and is the most versatile and cost effective plastic in comparison to other thermoforming and polyolefin materials (Essabir et al., 2013). It has good impact strength, surface hardness, dimensional stability and excellent abrasion resistance. Polypropylene is resistant to a wide variety of acids, alkalis and solvent solutions (Beckermann and Pickering, 2008; Cao et al., 2006). PP does not present stress-cracking problems and offers excellent electrical and chemical resistance at higher temperatures (Jahani and Ehsani, 2009).

Cellulose fibres are selected as reinforced filler for thermoplastics due to their superior mechanical properties and lower price compared to the synthetic fibres (Sanschagrin et al., 2011). The physical properties of fibre reinforced composite materials depend on the ability of the polymer matrix both to transmit stresses to the fibre reinforcement and to protect the fibres from damage (Li et al., 2007). To promote these characteristics and many others, filler treatment protocols, such as alkali, use of coupling agents and compatibilisers have been reported to improve interfacial adhesion (Faola et al., 2013; Husseinsyah et al., 2016; Oladele et al., 2010, 2019). Other than these approaches, high temperature calcination.
and pyrolysis can remove many of the amorphous, organic and hydroxyl groups present in natural fillers, hence it is expected that these techniques can lead to an improved interfacial adhesion between the fillers and the polymer matrices (Oladele et al., 2019).

Li et al. (2007) made a comprehensive study on various types of chemical treatments applied for the surface modification of the natural fibres where it was revealed that each treatment has its own advantages and disadvantage. Also, of the various chemical methods used, alkali was found to be the most economical and easier way of achieving improved properties while Alsahheed et al., (2013) found that higher concentration of chemical absorption is high which leads to poor wettability and disadvantage. Also, of the various chemical methods used, alkali was found to be the most economical and easier way of achieving improved properties while Alsahheed et al., (2013) found that higher concentration of chemical properties that will stand the test of time, it is necessary to consider in order to obtain a good fibre reinforced composites is the adhesion between the matrix and the fibre at the level of production. Due to the presence of a hydroxyl group in natural fibres, the affinity to moisture absorption is high which leads to poor wettability and weak interfacial bonding between the fibres and the matrices over a period of time.

In order to develop composites with better mechanical properties that will stand the test of time, it is necessary to consider hydrophobicity in the fibres by suitable chemical treatments. The effect of alkali on the cellulose fibre is a swelling reaction, during which the natural crystalline structure of the cellulose relaxes and hence, there is a lattice transformation (Oladele et al., 2019). The work by Faola et al. (2019) in which the influence of treated yam stem particulate on high density polyethylene composites was carried out revealed that reinforcement with optimum value was obtained at 20 wt%. This composition produces the samples with the best results in tensile, hardness, impact, flexural, TGA and XRD which implies that best results can be obtained by incorporating this amount of the particle into the matrix.

Hence, this research was carried out to optimise the potentials that are inherent in yam stem fibres after harvesting the yam tuber by investigating the influence of the treated particles on the properties of polypropylene. The vegetable fibre was selected and used in order to assess the suitability or otherwise of the agro waste from this geological zone for bio-filled polymer composites.

2. Materials and Methods

2.1 Materials

The materials used for this work were white yam (Dioscorea rotundata) stem obtained from farmland located in geographical coordinates of latitude 7° 36’ 95” North and longitude 4° 42’ 90” East, Ilesa West, Osun State, Nigeria which serves as the reinforcement and polypropylene supplied by SASOL Polymers South Africa. The polypropylene has a melt flow index (MFI) of 8.5 g/10 mins and a density of 0.907 g/cm³. Other materials used were maleic anhydride grafted polypropylene (MAPP) G-3003 which serves as a compatibiliser for polypropylene supplied by Eastman Chemical Company, in South Africa while sodium hydroxide (NaOH) was sourced from Pascal Scientific Laboratory, Akure, Ondo State, Nigeria.

2.2 Methods

2.2.1 Fibre Extraction and Preparation

Yam fibre was obtained from the stem of harvested white yam tuber. The stem was removed from the stick when the stem has dried with the use of a cutlass. The yam fibre was washed thoroughly with distilled water to remove any adhering undesired soil and sun-dried. This was further chopped into short fibres of 8-10 mm length using pen knife.

Parts of the chopped yam stem fibre were chemically treated with 1M NaOH solution at a temperature of 70 °C for 2 hours in a shaker water bath while some were left as untreated sample. Both treated and untreated fibres were washed and rinsed with distilled water until pH of the rinse solution stabilised at 7. The fibres were later oven dried for 48 hrs at 65 °C before they were pulverised. Both the treated and untreated chopped yam stem fibres were pulverised using domestic marlex grinder followed by sieving with sieve shaker from where particle sizes of <150 µm were collected and used.

2.2.2 Compounding and Production of Composite Materials

The pulverised treated and untreated yam stem particles were oven treated at 65 °C for 24 hours to further remove the moisture content before impregnating with polypropylene. Predetermined proportions of polypropylene, yam stem particle (YSP) and MAPP were compounded. Individual mixed samples were melt-blended together using twin-screw extruder at a Jone high-speed tumbler mixer of 40 rpm at 170-190 °C for 10 minutes to obtain homogeneous mixture. The extrudates after air cooled were granulated into pellets with a guillotine grinding machine and oven dried at 65 °C for 24 hours. The pellets were poured in a rectangular metallic
mould of dimension 150 x 100 x 4 mm and were compressed with Carver laboratory press maintained at 190 °C under a pressure of 10 tons for 10 minutes. The composites produced were immediately transferred to a cold press to cool down to room temperature.

2.2.3 Determination of the Yam Stems Particulate Constituents

The Goering and Van Soest procedure for the determination of total Neural Detergent Fibre (NDF) was used to test for lignin, cellulose and hemicellulose so as to ascertain the efficiency of each of the treatment given to the particulates in the removal of lignin and hemicellulose and retention of cellulose which the fibre needed for the reinforcement (Husseinsyah et al., 2016). The main effect of washing a natural fibre with a mild alkaline solution is the removal of waxes, hemicelluloses and a partial removal of the lignin present on their surface. This exposes the cellulosic content of the particulate for proper bonding of the matrix with the particulate. Also, Faola et al. (2013) reported that chemical treatment of fibres with potassium and sodium hydroxide is effective for removing the lignin and hemicellulose and retention of the cellulose.

2.2.4 Testing and Structural Characterisation of Samples

Composite samples were prepared for hardness, tensile, flexural, impact toughness tests and morphological examination in order to study the behaviour of the composite in service condition.

2.2.5 Tensile Test

A dumbbell-shaped cutter was used to punch out the tensile specimens from the composite samples. The test was carried out using Instron Universal Tensile Testing Machine in accordance with ASTM D638-10 standards. The test piece, which is of gauge length 25 mm, was fixed at the edges of the upper and lower grip of the machine and the test commenced. As the test piece is extended, a graph is plotted automatically and important tensile properties data were recorded. The load applied was 25 kN at a crosshead speed of 5 mm/min. Six pieces were tested for each sample from where the average value was taken.

2.2.6 Hardness Test

The hardness of the neat polymer and composites were measured with the aid of micro-hardness tester, model 900-390, in accordance with ISO 868:2008 standards. This machine measures the resistance to penetration by measuring the depth of impression. The test was carried out by indenting the sample with the instrument at a test load of 50 g, dwell time of 10 seconds and X50 magnification before taking the reading that was displayed on the monitor. Six values were taken for each sample, and the average was taken as the representative value.

2.2.7 Flexural Test

Flexural test was carried out using Tensometric Universal Testing Machine in accordance with ASTM D790. Each sample of dimension 150 x 50 x 4 mm cut out from composite samples was firmly mounted on the Tensometric machine and as the sample stretched, the computer generates the graph as well as the desired parameters. Three samples were tested from where the average value was determined.

2.2.8 Impact Test

The test was carried out on a Charpy impact testing machine (Instron CEAST 9050) in accordance with ISO179 standard. Samples cut into dimension 80 x 10 x 2 mm were placed horizontally on the machine with the notched surface directly opposite the swinging pendulum. The initial reading of the sample gauge length and the thickness were entered into computer system attached to the machine and the machine was switched on. The pendulum of the machine swung freely through an angle of 150 o and fractured the sample. Six (6) specimens were tested from where the mean value was evaluated.

2.2.9 Thermogravimetric Analysis of the Particles and Composites

Thermogravimetric analysis (TGA) was used to measure the weight loss of the particles as a function of rising temperature. It was carried out on the particles as well as the particulate reinforced PP composites developed to measure the level of weight loss as a function of increasing temperature, over a temperature range of 100-600 °C at a rate of 5 °C/min performed under nitrogen atmosphere with nitrogen gas flow rate of 60 mL/min.

2.2.10 X-ray Diffraction Analysis of Particulate and Composites

X-ray diffraction analysis was carried out on the particles and the composites produced. Each of the treated and untreated particles was compressed into disks using a cylindrical steel mould of (Ø = 15 mm) with an applied pressure of 20 MPa. A Phillips X-Pert diffractometer fitted with a ceramic X-ray diffraction tube was used to determine the crystallinity. The diffracted intensity of Cu Kα radiation (wavelength of 0.1542 nm) was recorded between 5° and 40° (20 angle range) at 40 kV and 40 mA.

2.2.11 Scanning Electron Microscope Observation

Field Emission Scanning Electron Microscope of Model JEOL JSM-7600F was used for the morphological characterisation of the composite sample surfaces. This was carried out to investigate the miscibility of the particles with the matrix at the fractured surfaces.
Samples cut with bench vice were cleaned thoroughly, air-dried and glued on sample holder before coated with thick irradium in JEOL sputter ion coater and observed. The magnifications and kV rating used for the Scanning Electron Microscopy (SEM) images presented in this research are X100 and 25.0 kV respectively while Secondary Electrons (SE) were used to produce the SEM Images.

3. Results and Discussion

Figure 1 shows the proportions of the lignin, hemicellulose and cellulose for the treated and untreated yam stem particles. It was revealed that untreated yam stem particulate has higher value of lignin and hemicellulose as compared with the treated. Pre-treatment of natural fibres was due to their hydrophilic nature as they are derived from lignocellulloses which contain strongly polarised hydroxyl groups making their rate of water absorption to be high. The high water absorption rate leads to the degradation of the fibres and the fibre-matrix interface resulting in loss of mechanical performance.

Since the lignin retains water in the fibres therefore, to reduce the water absorption rate of composite materials, the lignin content of the fibre has to be reduced (Susheel et al., 2009). This could be achieved by pretreatment of the fibre with chemicals that can help in reducing the lignin content. Based on this, the lignin content was reduced by 32% after treatment. Also, during chemical treatments, constituents like hemicellulose were hydrolysed by the action of alkaline solutions (Husseinsyah et al., 2016). In this research, the treatment causes the reduction in hemicellulose by 74%. The cellulose was reduced by 5% after the treatment which implies less reduction in the cellulose content of the fibre compared to other constituents. Therefore, sodium hydroxide solution is effective for the removal of lignin and hemicellulose and, the retention of cellulose which is the required constituent for the reinforcement. However, from Figure 1, other constituents like pectin, wax and ash that make up for the 100% were not determined.

Figure 2 shows the thermogravimetric analysis of the treated and untreated yam stem particles. From the results, thermal decomposition of the particles as measured by weight loss and temperature was observed between 100-600 °C for both treated and untreated yam stem particles. Weight loss in fibres occurs due to the decomposition of cellulose, hemicellulose and lignin constituents during heating (Methacanon et al., 2010; Kabir et al., 2013).

It was observed that the untreated materials start to lose weight at lower temperature than the treated materials. This was attributed to the reduction of the higher moisture content of untreated yam stem particles that were present in the hemicelluloses. The decomposition process was very mild until around 250 °C for both the treated and the untreated particles. However, a large weight loss was observed at temperatures above 250 °C up to 400 °C. At these temperatures, the cellulose was entirely decomposed due to the breaking down of its molecular structures. The decomposition temperature of the treated was higher than that of the untreated. This was due to the fact that most of the cellulose structure in treated fibre was crystalline. The cellulose was strong and has high resistant to hydrolysis due to the presence of strong intramolecular and intermolecular hydrogen bonding that required higher energies to be broken down. Therefore, the treated fibre was more thermally stable than the untreated in agreement with works of Place et al. (2009) and Tajeddin et al. (2009). This also corresponds with the findings of Kim and Eom (2001) and Nair et al (2001) that removal of thermally unstable constituents such as hemicellulose and pectin could result in higher decomposition temperatures, and hence, greater thermal stability. It was also observed that the untreated particles degrade at lower temperatures compared with the treated. This was also reported by Beckermann and Pickering (2008) that; untreated fibre degrades at lower temperatures due to the presence of thermally unstable fibre constituents such as hemicelluloses and pectins,
whereas the alkali treated fibre is more thermally stable due to the removal of these constituents.

Figure 3 shows the X-ray diffractograms of the treated and untreated yam stem particles, while Table 1 shows the crystallinity index. The low intensity peak represents the amorphous phase \( I_{am} \) and the higher intensity peak represents the crystalline phase with the crystallographic plane (002). The plot exhibits two prominent peaks obtained at 2θ values of 32° and 35°. It was observed that the crystallinity index of the treated particle was higher than that of the untreated. Similar results were reported by Mangesh and Akshay (2017) where the findings were attributed to removal of amorphous materials such as hemicellulose, lignin and pectin by the alkali treatment. This invariably results in close packing and stress relaxation of the cellulose chains, hence, an increase in the degree of crystallinity.

![Figure 3. X-ray Diffractograms of Treated and Untreated Yam Stem Particulate](image)

Table 1. Crystallinity Index Values for both Treated and Untreated Yam Stem Particulate

<table>
<thead>
<tr>
<th>Particulate</th>
<th>( I_{am} ) (2θ = 32.5)</th>
<th>( I_{002} ) (2θ = 35.5)</th>
<th>Crystallinity index %</th>
</tr>
</thead>
<tbody>
<tr>
<td>Treated</td>
<td>2991</td>
<td>11250</td>
<td>73.4</td>
</tr>
<tr>
<td>Untreated</td>
<td>2691</td>
<td>9250</td>
<td>70.9</td>
</tr>
</tbody>
</table>

![Figure 4. Ultimate Tensile Strength of Particulate Reinforced Polypropylene Composites and the Control](image)

However, crystallinity index is useful only on a comparison basis as it is used to indicate the order of crystallinity of the crystalline regions (Deshmukh et al., 2010). The crystallinity index \( Crl \) of the treated and untreated yam stem particles was calculated using the formula in Equation (1).

\[
Crl(\%) = \frac{(I_{002} - I_{am})}{I_{002}} \times 100
\]

Where, \( I_{002} \) and \( I_{am} \) are the peak intensities of crystalline and amorphous materials, respectively. \( I_{002} \) is the maximum intensity of diffraction of the (0 0 2) lattice peak at a 2θ angle 35.5° and \( I_{am} \) is the intensity of diffraction of the amorphous materials which is taken at a 2θ angle 32.5° where the intensity is at a minimum (Essabir et al., 2013).

Figure 4 shows the ultimate tensile strength of treated and untreated yam stem particulate reinforced polypropylene composites. Tensile strength indicates the ability of a material to withstand forces that pull it apart as well as the capability of the material to stretch prior to failure. It was observed from the plots that; the composites samples had improved tensile strengths than the control sample. However, treated samples showed better enhancement compared to the untreated ones. This may be attributed to the chemical treatment given to the particle which was effective in increasing the tensile strength of the treated particulate reinforced PP composite samples. Though, both treated and untreated YSP/PP composites samples showed a similar trend with increasing in tensile strength from 10 to 30 wt.% reinforcement content. Within this range, only treated YSP/PP and 30 wt.%. untreated YSP/PP composites have enhanced strength more than the control sample. Thereafter, a sharp decrease was observed in the tensile strength of the composite samples when the reinforcement content was increased to 40 wt.%. Hence, treated YSP/PP composite sample reinforced with 30 wt.% gave the optimum result with a value of 34.82 MPa while the control sample is 33.60 MPa.

Tensile modulus for the control and YSP/PP composites are illustrated in Figure 5. Similar trends with that of Figure 4 were observed with slight difference. From the results, only treated YSP/PP composites reinforced with 30 - 40 wt.% possess enhanced tensile modulus compared with the control sample. The highest was from 30 wt.% reinforced sample with a value of about 1454.02 MPa slightly followed by sample with 40 wt.% having a value of 1418.13 MPa while the control sample was with a value of 1381.09 MPa. This increase in modulus within this range was likely due to the fact that the filler was well dispersed within the matrix. Hence, the optimum particle loading was obtained at 30 wt.% reinforcement. This corresponds with the findings of Beckermann and Pickering (2008) that increase in tensile modulus of the kenaf treated fibre reinforced composites improves over the untreated fibre reinforced composites.
due to alkaline treatment attributed to improved bonding between the fibre and matrix.

Figure 5. Tensile Modulus of Particulate Reinforced Polypropylene Composites and the Control

Figure 6 shows the hardness properties of both the treated and untreated YSP reinforced polypropylene composites. Here, an increase in hardness strength of treated YSP reinforced polypropylene composites was observed in all the composites. The hardness increases from 10 to 20 wt.% before it starts to decrease from 30 to 40 wt.%.

Figure 6. Hardness Properties of Particulate Reinforced Polypropylene Composites and the Control

Figure 7 shows the impact properties of the treated and untreated YSP reinforced polypropylene composites. In this figure, a decrease in impact strength from 10 wt.% to 40 wt.% was observed for treated polypropylene thus, making 10 wt.% to have the highest impact value while for untreated polypropylene, impact strength increases from 10 wt.% to 20 wt.% particulate loading before a decrease in impact strength was observed from 30 wt.% to 40 wt.%. The decrease in impact energy for an increase in filler content for the treated samples may be attributed to the weak interfacial interaction between the filler and the matrix material for higher filler content, similar behavior of the composites samples was also reported by Jahani and Ehsani (2009). However, treated polypropylene composites give the highest impact strength than the untreated ones.

Figure 7. Impact Properties of Particulate Reinforced Polypropylene Composites and the Control

Flexural strength is the ability of the composite material to withstand bending forces applied perpendicular to its longitudinal axis. Figure 8 shows the flexural strength of the treated and untreated YSP reinforced polypropylene composites. It was observed that treated polypropylene composites show an increase in flexural strength from 10 to 20 wt.% before a steady decrease was observed from 20 to 40 wt.% particulate loading while the untreated polypropylene composites show a decrease in flexural strength with increasing in particulate loading from 10 to 40 wt.%. However, treated polypropylene composites have higher flexural strength value of 40.58 MPa compared to the untreated polypropylene composites and neat sample with a value of 36MP and 19 MPa, respectively.

Figure 8. Flexural Strength of Particulate Reinforced Polypropylene Composites and the Control
Flexural modulus for neat, treated and untreated YSP polypropylene composites are illustrated in Figure 9. The flexural modulus value increases linearly with increasing in reinforcement content from 10 to 30 wt.% for treated polypropylene composites; thereafter a noticeable decrease was observed at 40 wt.% while the untreated polypropylene composites show a slight decrease with increasing in particle loading up to 20 wt.%. However, polypropylene composites with 30 wt.% reinforcement content have the highest flexural modulus for both the treated and untreated with a respective value of 965 and 693 MPa. The increase in flexural modulus is governed by the fact that the filler gives good reinforcement with the matrix with well dispersed particulate. Previous report showed that, increase in modulus was due to the presence of compatibiliser and the high surface area of the filler leading to increase in interfacial adhesion (Faola et al., 2013). However, after 40 wt.%, the particles get agglomerated while processing the sample. Therefore, the dispersion becomes poor and the modulus decreases.

Figures 10 and 11 show the surface morphology for the yam stem particulate (YSP) from where it was noticed that the chemical treatment has actually removed some of the lignin that may hinder proper interfacial adhesion as a smooth constituent, therefore, leaving the surface of the treated yam stem particulate wrought (see Figure 10) compared to the untreated particulate (see Figure 11) which has its surfaces smooth. Composites from untreated particles were observed to possess some features like agglomerations and voids as shown in Figure 11 which were responsible for the poor mechanical properties in the untreated YSP composites compared to the treated YSP based composites.

4. Conclusion
Treated and untreated yam stem particulate reinforced polypropylene composites were developed and characterised. The results obtained from the mechanical and morphological analysis carried out show that; chemical treatment was found to be effective for the reduction of lignin and hemicellulose as well as retention of the cellulose constituents of the yam stems particles. Hence, composites developed from treated yam stem particulates perform better than the untreated composites due to the presence of higher proportion of cellulosic fibre and adequate surface roughness that promote good interfacial adhesion between the filler and the matrix.

However, both composites gave better results than the unreinforced samples from polypropylene in all the tests carried out. The reinforcement with optimum value was obtained at 20 wt.% yam stem particulates addition. Hence, the use of this agro waste in polymer composites development will promote the production of green composites which are in current needs.

References:


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A Study on Mechanical Properties of Aluminum-Graphite and Aluminum-Graphite-Zircon Hybrid Composites

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Abstract: Aluminum-based Graphite (Al-Gr) composites have been used in automotive and industrial applications owing to their good mechanical and physical properties compared to homogenous materials. Although Al–Gr composites have reportedly showed good compressive behaviour; however, there is the need for further improvement on the mechanical property, hence the need of Aluminum Hybrid composites. An emerging material among the growing list of Aluminum Hybrid Composites is the Al–Graphite-Zircon (Al-Gr-ZrSiO4) composites whose mechanical properties have been evaluated but for few reported investigations on its compressive behaviour. In this study, the mechanical behaviour of Al-Gr and Al-Gr- ZrSiO4 composites under compressive loading are evaluated. Using reinforcement of 4 and 8 wt.% each in Al-Gr and Al-Gr- ZrSiO4 composites samples produced by stir casting and subjected to standard heat treatment, compression strength, modulus of compression, compression strain to failure, impact strength and micro hardness including density were evaluated. The results obtained show reduction in impact strength and compressive modulus with increased weight fraction of reinforcement while other mechanical properties improve significantly with increase in wt. % fraction of reinforcement. For the Al-Gr composites, all the mechanical properties tested decreases with increase in weight fraction of the graphite reinforcement with exception of hardness which increase marginally.

Keywords: Aluminum, Zircon, Graphite, Hybrid Composites, Compressive Strength, Stir casting

1. Introduction

The use of Graphite and Zircon is an emerging idea to reinforce Aluminum owing to their individual reinforcement properties. Graphite has low density, good compressive strength and thermal stability as a result of its high melting temperature, high strength to mass ratio, as well as good solid lubrication properties (Gaitonde et al., 2012; Biswas and Rohatgi, 1983). Hence, Al-Gr composites are characterised to have properties making them suitable in various applications having low density and good thermal properties, such as base plates or coolers for power semiconductor devices and heat sinks to mention a few. Good compression strength and wear resistance application for piston and cylinder liner materials (Shanmughasundaram and Subramanian, 2013; Hazim et al., 2015).

Also, Zircon is known to possess high hardness, strength, density, fracture toughness, chemical inertness and wear resistance with low coefficient of thermal expansion which makes its use as reinforcement for Aluminum to produce materials for applications in automobile industries, recreational products and in construction company (Raju and Ramanurthy, 2018; Sucitheran et al., 2013; Okafor and Aigbodion, 2010). Furthermore, the two reinforcements have been used independently with other reinforcements to improve on the mechanical properties of Aluminum (Bodunrin et al., 2015; Baghchesara and Abdizadeh, 2013). Expectedly, the use of two reinforcements namely; Zircon and Graphite to produce hybrid composite could offer a better combination of the individual properties.

A comparative study on the synthesis and characterisation of Al 7075 – ZrSiO4 and Al 7075 – Flyash – Gr was reported (Krishnan et al., 2018). Higher hardness and density were observed in Al-Gr-ZrSiO4 composites compared to Al 7075 – Flyash – Gr composites due to the presence of ZrSiO4 in the former. The microstructure, micro hardness and compressive behaviour of Aluminum alloy (ADC – 12) reinforced with Titanium Carbide and Zircon was studied (Yandav et al., 2015). It was observed that the optimum combination of properties was exhibited by Aluminum reinforced with equal amounts of both reinforcements with compressive properties and micro hardness all showing improvement. An investigation on the mechanical properties of Al5083 composites reinforced with Zircon and Rice husk particles produced using stir casting was also carried out (Dhileepan et al. 2018). The
weight fraction of the Rice husk was varied while that of Zircon was fixed. The result showed that the addition of fixed amount of Zircon increased the hardness of the hybrid composites when compared to the composite devoid of ZrSiO₄.

Similarly, increase in the hardness of Aluminum (Al6011) reinforced with graphite and Zircon produced by stir casting technique and subjected to T6 heat treatment was reported (Gopi et al., 2013). The increase in the hardness was attributed to addition of Zircon because Graphite was fixed for the range of weight fraction investigated. A review on the effect of Zircon on the mechanical properties of Aluminum composites was carried out and it was concluded that Zircon can be successfully used to improve on the ultimate tensile strength and hardness of Aluminum (Thandallam et al., 2015). Also, the influence of Graphite on the mechanical behavior of Al7075 – Al₂O₃ – Gr hybrid composite was investigated (Baradeswaran and Perumal 2014). The hardness, tensile strength, flexural strength and compression strength of the composites were observed to increase with increasing weight fraction of the reinforcements. Furthermore, increase in hardness of Aluminum hybrid composites containing Silicon Carbide and Graphite particles with a reducing Graphite weight fraction has also been reported (Velmurugan et al., 2011; Ramnath et al., 2018).

While few works have investigated the mechanical behaviour of Al – Gr – ZrSiO₄ hybrid composite, its compressive behaviour under compressive loading in applications such as pistons for light engines using low cost materials and comparative study of its properties to that of Al – Gr composites have not been fully studied. Therefore, in this study, investigation on the compressive behaviour of hybrid Al – Gr – ZrSiO₄ and Al – Gr composites using 4 and 8wt.% reinforcement for each composite was carried out including hardness, impact strength and density.

2. Methodology
2.1 Materials and Processing

The Aluminum alloy used is an Al 6011 ingot with chemical composition shown as in Table 1 and obtained from Tower Aluminum, Ota, Ogun State, Nigeria. The ingot was cut into pieces of about 20 cm thickness and 10 cm in length with the use of a power hack saw and further sheared into smaller irregular shaped pieces with the use of a shear cutter to enable easier weighing of smaller quantities. Graphite particles (reinforcement) and the wetting agent (magnesium) were purchased from certified local vendor in Lagos, Nigeria. Graphite and Zircon were collected in lump and particle form respectively and subsequently milled to 100 microns. The weight of reinforcements (4 and 8wt. %) measured for both the Al–Gr and Al–Gr–ZrSiO₄ composites was weighed using an electronic weighing balance.

The choice of wt. % used in this work is based on previously reported study on the tendency of mechanical properties of stir cast Al – Gr and Zircon-based Aluminum hybrid composites to increase marginally up to 4wt. % and a tendency of deterioration beyond 7wt. % (Krishna et al., 2017; Gopi et al., 2013; Ramesh et al., 2009). For the two Al – Gr composites, Graphite weight is 4 and 8 wt. % of the total weight of the charge for melting. Also, for the two Al–Gr–ZrSiO₄ hybrid composites, the reinforcement (combination of Gr and ZrSiO₄ derived from their density ratio) is 4 and 8 wt. % of the total charge for melting. The Al–Gr–ZrSiO₄ containing 4wt.% of reinforcements has 2.7wt.%Zircon and 1.3wt.%Gr while the Al–Gr–ZrSiO₄ containing 8wt.% of reinforcements has 5.4wt.%Zircon and 2.6wt.%Gr. The derivation of the wt. % reinforcement of the hybrid composites for 4 and 8 wt. % using density ratio is shown in Equation (1). The formulation of Al – Gr composites and Al – Gr – ZrSiO₄ composites is showed in Table 2.

\[
W_c = W_m + W_r
\]

Where;
\[
W_i = (\rho_{gr} \cdot \varepsilon) + (\rho_z \cdot \varepsilon)
\]

Where; 
\(W_i\) is the total weight of hybrid reinforcement measured. \(\rho_{gr}\) and \(\rho_z\) are the density ratios of Graphite and Zircon respectively. 
\(\varepsilon\) is the weight fraction (%) of the Hybrid reinforcement in the composite.

Table 1. Chemical Composition of the Aluminum Alloy

<table>
<thead>
<tr>
<th>Element</th>
<th>Al</th>
<th>Si</th>
<th>Fe</th>
<th>Cu</th>
<th>Mn</th>
<th>Mg</th>
</tr>
</thead>
<tbody>
<tr>
<td>Composition (%)</td>
<td>97.91</td>
<td>0.4835</td>
<td>0.7627</td>
<td>0.0951</td>
<td>0.0775</td>
<td>0.3052</td>
</tr>
</tbody>
</table>

Table 2. Formulation of Aluminum Composites Samples

<table>
<thead>
<tr>
<th>S/N</th>
<th>Al–Gr composites</th>
<th>Al–Gr–ZrSiO₄ composites</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>96wt.% of Al + 4wt.% of Graphite</td>
<td>96wt.% of Al + 1.3wt.%Gr + 2.7wt.%ZrSiO₄</td>
</tr>
<tr>
<td>2.</td>
<td>92wt.% of Al + 8wt.% of Graphite</td>
<td>92wt.% of Al + 5.4wt.%ZrSiO₄</td>
</tr>
<tr>
<td>3.</td>
<td>100% Aluminum</td>
<td></td>
</tr>
</tbody>
</table>

The composites were produced with the use of a carefully designed electric resistance furnace via stir casting. Stir casting is a low cost, time saving and easy to use liquid processing technique in metal casting where matrix alloy is heated beyond melting temperature and reinforcement particles are introduced with consequent stirring for uniform distribution of the particles resulting in
near net shape cast. Before charging, the reinforcements and wetting agent as well as the mould are preheated to 300°C, essentially to reduce moisture which can cause faster cooling and thus lead to deterioration in the quality of the samples to be cast. The carefully designed and constructed rectangular electric furnace of dimension 35cm x 35cm x 36cm built by assembling grooved low density refractory bricks (light bricks) and electric resistant wire (Nichrome 80) as source of heat energy. The refractory bricks were obtained from Lightning Vapes, the United States of America (USA). The light bricks are 23 cm x 7 cm x 12 cm in dimension, with a density of 0.763g/cm³. The Nichrome wire has a diameter of AWG 18 (0.102 cm) with maximum operating temperature of 1180°C and resistance of 20 Ω.

The Aluminum matrix melts in the electric furnace at about 750 °C after which the reinforcement is introduced with the wetting agent into the melt, stirred for about 30 seconds and poured individually into a finger mould that have five (5) holes each of depth 135 mm and diameter 15 mm. After cooling, the cast is removed from the mould, and to reduce casting defects, such as micro segregation and porosity, homogenise reinforcement particles and improve hardness, the composites, as well as the unreinforced alloy were subjected to T6 heat treatment. The T6 heat treatment is a standardised sequential heat treatment procedure involving solution heat treatment followed by water quenching and subsequently age hardening. The composites were first homogenised at 530°C for 3 hours using a Lab Science box resistance furnace with Model dfw - 7000 and subsequently quenched in water to retain the high temperature microstructure. After quenching, the samples were returned into the electric resistant furnace for artificial ageing at 180°C for 5 hours. The cast samples were subsequently machined to the specification for the various mechanical test that was carried out.

Figure 1. (a) Cast Samples, (b) Mould

2.2 Microstructural Analysis

2.2.1 Scanning Electron Microscopy (SEM)

Scanning electron microscopy of test samples was carried out using a High-Performance Scanning Electron Microscope (SEM) (VEGA 3 TESCAN) at accelerated voltage of 20KV to reveal the details of the microstructure of the composite samples and defects. The micrographs are consequently obtained using Backscattered Electron (BSE) mode with magnification up to 2000X. Prior to microscopic examination, samples were prepared by grinding with emery papers with grit size 60, 150, 220, 320, 400, 600, 800 and 1200, in successive order and mounted on a conductive carbon imprint left by the adhesive tape prepared by placing the samples on the circular holder and coated for 5 minutes to enable conduction.

2.2.2 X-Ray Diffractometer (XRD)

To identify the reinforcement in the matrix alloy, XRD of the stir cast composites was carried out. The XRD of the samples was performed using PW1710 Philips X-ray diffractometer with Cu, Ka radiation at 40 kV and 40 mA and were analysed using PANalytical X’Pert High Score software for phase search-match. Samples were exposed to a monochromatic Cu Ka radiation (k = 1.5406 Å), operating at 40KV and 40mA. The samples were registered in a zero-background sample holder to avoid external background interferences. The diffractograms were registered in the range of 5° to 90° (2θ) in a step scan mode of 0.0170 at a scan step time of 1.00s.

2.3 Composite Density Determination

The density of composites was obtained by measuring weights of samples in air and dividing this by the volume of water being displaced using the Equation (3). Weights of samples were measured using an electronic weighing balance while samples were immersed into a water containing cylindrical flask to measure the volume of water displaced.

\[
\text{Density} = \frac{\text{Mass of sample}}{\text{Volume of water displaced}} \quad (3)
\]

2.4 Mechanical Tests

Compression test was carried out on test samples with dimension 15 mm diameter and 25 mm height using a Universal Tensile Testing machine (Instron Universal Tensile Testing Machine of Model 3369). Other mechanical properties obtained from the data include, modulus of compression and compression strain to failure. Hardness test was performed using a SIOMM Vickers hardness tester with model HVD – 1000IS. Hardness test samples were prepared by grinding cut samples with emery paper of grit sizes 60, 150, 220, 320 and 400, in successive order to allow for proper micro hardness test indentation. The samples were indented for a dwell time of 10 s with a load of 200 g. The indentation was taken in three different places for each sample and the average results recorded.

Impact test was performed to measure the energy absorbed before fracture of the composite. An Avery Charyp Impact Testing Machine with model number E67424/4 was used. The impact testing machine has a striking force of 298 Nm and a striking velocity of 5 m/s. Impact test samples were prepared by machining composites to diameter of 10 mm.
and height of 55 mm while a notch of 2 mm at angle 30° at the center of the samples.

3. Results and Discussion

3.1 Microstructural Analysis

3.1.1. Scanning Electron Microscopy Evaluation of Al-Gr and Al-Gr-ZrSiO₄ Composites

Figures 2 and 3 refer to the microstructure of Al-Gr composites while Figures 4 and 5 show that of Al-Gr-ZrSiO₄ hybrid composites. All the figures show segregation of Graphite (as it is observed to be poorly dispersed in the matrix), while Figures 4 and 5 show Zircon in a much more dispersed form in the microstructure. Segregation of Graphite in Aluminum can be attributed to its low density and poor settling during stirring operation during casting. Segregation in Al-Gr composites has also been reported in other works (Sharma et al., 2015; Sidharta et al., 2018). It can be observed that segregation in the microstructures of both composites tend to increase with increased weight fraction. This was attributed to wettability of the Graphite particles to the matrix to be more impaired at high weight fraction (Saheb, 2011). Thus, the powders begin to cluster and create uneven graphite distribution in the matrix.

3.1.2 X-Ray Diffraction Evaluation of Al-Gr and Al-Gr-ZrSiO₄ Composites

XRD peaks indicate the presence of phases present in the composites. Figures 6(a) – (c) show the XRD spectra of aluminum, graphite and zircon. Figure 6(a) indicates the peaks of aluminum at 2θ = 38.50°, 44.13/60°, 65.12°, 77.92° and 82.46°. Figure 6(b) indicates the peaks of graphite at 2θ = 26.67°, 44.95°, 54.48° and 77.60° while Figure 6(c) shows the peaks of zircon. The peaks of the zircon phase are observed at 2θ = 26.96°, 38.47°, 43.26°, 54.95°, 62.76°, 68.65°, 75.54° and 79.11°. The identification of quartz phase in the XRD is as a result of presence of quartz as an impurity in the zircon material.

The XRD of Al-Gr and Al-Gr-ZrSiO₄ hybrid composites are presented in Figure 7. This study only revealed the phases of interest in the XRD as the aim of the XRD is to investigate the presence of the reinforcement in the matrix alloy. Figure 7(a) shows the peaks of aluminum
and graphite phases. Also, Figure 7(b) shows the peaks of aluminum, zircon and graphite phases in the composites.

![Figure 7. XRD of (a) Al-Gr composites, (b) Al-Gr-ZrSiO₄ hybrid composites (α = aluminum, g = graphite, z = zircon).](image)

3.2 Influence of Reinforcement on Density of Al-Gr and Al-Gr-ZrSiO₄ hybrid composites.

Specific gravity or lightness (low density) is an attractive property of composites and are usually achieved by the introduction of low density reinforcements into the matrix. The resulting composites are less dense. In addition, changes in the density of matrix with the introduction of ceramic reinforcement can also be due to the thermal mismatch between the metal matrix and the reinforcement as a result of the differences in their coefficient of thermal expansion and the elastic properties (modulus) of the matrix and the reinforcement (Sharma, 2003).

As observed in Figure 8, there is a change in the density of Aluminum with the introduction of the reinforcements. The density of Al-Gr composites decreases with increasing weight fraction of Graphite. A reduction of 14% and 17% was observed in Al – 4wt. % Gr and Al – 8wt. % Gr composites respectively. This can be attributed to the lower density of Graphite, 2.2g/cm³ and the result is consistent with other similar works on Al-Gr composites (Sharma et al., 2015). Thus further increase in the wt. % of Graphite might lead to further reduction of density of the composites. However, for Al-Gr-ZrSiO₄ hybrid composites, the density increases with increasing wt. %. For Al – 1.3wt%Gr – 2.7wt.%ZrSiO₄ composite, the density is observed to increase by 6.53% (2.86 g/cm³) while for Al – 2.6wt%Gr – 5.4wt.%ZrSiO₄ to increase up to 8.75% (2.92 g/cm³) when compared to density of unreinforced Al 6011 at 2.68. The increase in density with increasing hybrid reinforcement can be attributed to the inherent high density of Zircon.

![Figure 8. Effect of Reinforcement on the Density of Al-Gr and Al-Gr-ZrSiO₄ hybrid composites.](image)

In a related work, the density of Al 7075 reinforced with Zircon and Graphite increases with increasing weight fraction (Krishna et al., 2017). This can be expected as the density of Zircon is 4.65 g/cm³ which is expected to impact on the density of the Al-Gr-ZrSiO₄ hybrid composites (Rino et al., 2013).

3.3 Influence of Reinforcement on Hardness of Al-Gr and Al-Gr-ZrSiO₄ hybrid composites

Hardness, often described as a measure of a material's resistance to surface indentation, may be thought of as a function of the stress required to produce some specific types of surface deformation. Hardness is a very important property to be considered in the application of AMC especially where resistance to deformation by means of abrasion is highly required. Hence, reinforcements are usually considered based on their inherent hardness property for such applications. This is because presence of reinforcements especially with high Mohs scale value aids the matrix alloy in resisting application of load by indentation which it transfers to the reinforcing particles. Thus, the higher hardness observed in the hybrid composite in Figure 9 compared to the Al-Gr composites can be attributed to the introduction of Zircon with higher Mohs scale hardness value (7) compared to Graphite (2) in the hybrid composite.

![Figure 9.](image)

In addition, the presence of Zircon is known to improve the bonding strength of the matrix – reinforcement interface, which is necessary for improvement of the hardness of composites (Ramnath, 2018). Furthermore, the
T6 heat treatment carried out on all the test samples have greatly impacted on the hardness property of the composites especially Al-Gr composites having a rather soft reinforcement (Fabrizi et al., 2018).

The hardness of Al-Gr composites increased with increase in weight fraction (see Figure 9). There was a slight increase of 4.5% in Al – 4wt. % Gr composites and a higher increase of 125% at Al – 8wt. % Gr composites. In a previous study, Satishkumar (2017) reported an increase in hardness of Aluminum with the incorporation of Graphite as reinforcement after subjecting composites to T6 heat treatment. For the Al – 1.3wt%Gr – 2.7wt.%ZrSiO₄ composite, the hardness increases to 975HV and 1219 HV for Al – 2.6wt%Gr – 5.4wt.%ZrSiO₄. This is an increase of 132% and 190%, respectively, over the unreinforced Aluminum alloy at 419.83 HV. Similar findings on the increase in hardness of Aluminum composites reinforced with ZrSiO₄ both as single reinforced and hybrid reinforced have been previously reported (Raju and Ramamurthy, 2018; Yaday et al., 2015; Gopi et al., 2013; Rino et al., 2013).

![Figure 9. Effect of Reinforcement on the Hardness of Al-Gr and Al-Gr-ZrSiO₄ hybrid composites](image)

### 3.4 Influence of Reinforcement on the Compressive strength of Al-Gr and Al-Gr-ZrSiO₄ hybrid composites

Compressive strength of a material is the maximum compressive stress that, under a gradually applied load, a given solid material can sustain without fracture. Its significance lies in the capacity of the material to withstand loads tending to reduce its size, as opposed to tensile strength, which withstands loads tending to elongate. Compressive strength of Aluminum is observed to increase with the addition of reinforcements in both Al-Gr and Al-Gr-ZrSiO₄ hybrid composites as observed in Figure 10. The increase in compressive strength of composites have been generally attributed to the reinforcements acting as barriers to dislocation movement arising from compressive loading on the matrix alloy. Thus, the mechanism responsible for increase in compression strength achieved is similar to that of hardness.

Hence, the two properties show improvement in composites as observed in Figures 8 and 9 (Ramesh et al., 2009). The compressive strength of Al – 4 wt. % Gr composites at and Al – 8 wt. % Gr are 200 MPa (191% increase) and 166 MPa (141% increase), respectively, compared to the unreinforced Aluminum alloy (69MPa), which is similar to the report by Ramesh et al. (2009). In addition to dispersion strengthening mechanism discussed above, increase in compressive strength of Al-Gr composites (200 MPa) compared to unreinforced Aluminum can also be attributed to the inherent compressive strength of Graphite (Hazim et al., 2015).

However, the higher compressive strength of Al – 4wt.% Gr composite compared to Al – 8wt.% Gr composite can be attributed to decline in segregation of Gr particles in the in Al – 4wt.% Gr composites as seen in Figure 2 compared to that of Al – 8 wt.% Gr composite shown in Figure 3. This may also be responsible for its superior strength over the hybrid composites. For the Al-Gr-ZrSiO₄ hybrid composites, the compressive strength increases with weight fraction in Al – 1.3wt.%Gr – 2.7wt.%ZrSiO₄ (163 MPa) and Al – 2.6wt%Gr – 5.4wt.%ZrSiO₄ (179 MPa), which is 136% and 159% higher than the unreinforced Aluminum alloy, respectively. The increasing weight fraction of hard ceramic material (Zircon) in the hybrid composites is responsible for the increase in compressive strength of the hybrid composite at higher wt.% as evident in Figure 9 compared to Al-Gr composite at same wt.%.

![Figure 10. Effect of Reinforcement on Compressive strength of Al-Gr and Al-Gr-ZrSiO₄ hybrid composites](image)

### 3.5 Influence of Reinforcement on the Compressive Strain to Failure

Compression strain to failure is a measure of the percentage reduction in size of a material at the point of failure. It is also an indication of the extent of resistance of materials to failure. Compression strain to failure is dependent on the compression strength of materials as the strength of a material determines its resistance to failure. The
consistency of the pattern of compression strength behaviour and compression strain to failure in Figures 11 and 12 for each of the composites shows the relationship between the two properties. At high wt. % of Graphite (Al – 8wt.% Gr composite), there is a reduction in the compression strain to failure compared to lower wt.% (Al – 4wt. % Gr composite). This might be attributed to the decreased resistance to compressive load (compressive strength) at point of failure of Al – 8wt. % Gr composite owing to increased segregation of Graphite in the composite compared to the segregation in Al – 4wt.% Gr composite.

This is not the case as observed in Al-Gr-ZrSiO₄ composites where compressive strain to failure increases with increasing wt. % The high compression strain to failure observed at higher wt. % (Al-Gr-ZrSiO₄ with 8wt.% reinforcement) of hybrid composites is attributed to the presence of hard Zircon particles offering resistance to failure when subjected to static deformation in compression mode at failure. The Al-Gr composites show significant reduction in size (see Figure 11) where it fails at 4wt. % reinforcement (160%) compared to its failure at 8wt. % reinforcement (114%). For the Al – Gr – ZrSiO₄ hybrid composites, the compression strain to failure is observed to increase with increase in wt. % of hybrid reinforcements. For Al – 1.3wt%Gr – 2.7wt.%ZrSiO₄, there is an increase of 130% over the unreinforced Aluminum alloy while for Al – 2.6wt%Gr – 5.4wt.%ZrSiO₄ the increase is 220%.

3.6 Influence of Reinforcement on the Impact Energy of Al-Gr and Al-Gr-ZrSiO₄ hybrid composites

Impact Energy is a measure of the amount of energy a material can absorb before brittle failure when dynamically loaded. Impact test are a measure of a given material’s toughness and can also be used to determine temperature-dependent brittle-ductile transition curve of a material (Ramesh et al., 2009; Sakthivei and Ramesh, 2013). Impact energy of the two composites (Al-Gr and Al-Gr-ZrSiO₄) are observed to reduce significantly when compared to the unreinforced matrix. This may be due to the presence of the reinforcement acting as stress concentration sites for crack propagation hence the deterioration in their (composites) ability to absorb energy at fracture when compared to the unreinforced matrix (Sozhamannan et al., 2012; Chawla and Chawla, 2006). Moreover, the T6 treatment of the composites which improves the hardness of the composite might have caused the reduction of impact energy. The hardening through ageing may cause embrittlement of the matrix – reinforcement interphase, which is expected to result in poor ability to absorb shock. The inverse relationship in the properties is evident in Figures 9 and 12 as Al-Gr composites show higher Impact Energy consistently than Al-Gr-ZrSiO₄ composites with the inverse being the case when compared with the hardness property. The higher Impact energy showed by Al – Gr composites might be attributed to the ‘softer’ nature of Graphite particles thus providing better plastic deformation unlike the harder Zircon particles.

However, Impact energy behaviour of the composites as observed in Figure 12 shows that Al-Gr composites shows a reduction of 44% and 53% for Al – 4wt. % Gr and Al – 8wt. % Gr composites respectively. Further reduction at higher wt. % of Graphite might be due to clustering of Graphite particles. A previous work reporting on the reduction of Impact Energy with introduction of Graphite, attributed the reduction to the clustering or micro segregation of Graphite particles (especially at higher wt. % above 4wt. %) as well as porosity (Sidharta et al., 2018). Al – 4wt. % Gr composite shows a reduction of about 80% (6.78J) while Al – 8wt. % Gr composite shows a reduction of 60% (13.56J) in Impact strength compared to the unreinforced Aluminum (33.91J). The Al – 2.6wt%Gr – 5.4wt.%ZrSiO₄ composite showed a higher resistance to impact force compared to Al – 1.3wt%Gr – 2.7wt.%ZrSiO₄ composite.
3.7 Influence of Modulus of Compression on Al-Gr and Al-Gr-ZrSiO4 hybrid composites

Modulus of compression is a material property that defines the degree of stiffness of materials as it predicts the resistance of a material to dimensional change in the linear regime of deformation of a material when subjected to compressive loading. Figure 13 shows the effect of Graphite and Zircon on the modulus of compression of Aluminum. The Graphite particles improved on the compressive modulus of Aluminum as Al-Gr composites shows an increase of 33% at 4wt.% reinforcement (8 GPa) and 3% increase at 8wt.% reinforcement (6 GPa). The Al – 4wt.% Gr composite shows good compressive modulus and compressive strength (see Figure 9), which shows that Graphite provides good resistance to both plastic deformation and elastic deformation in the matrix at that weight fraction. However, the Al-Gr-ZrSiO4 composite shows a general reduction in compressive modulus with increasing weight fraction of reinforcement.

![Figure 13. Effect of Reinforcement on Compression Modulus of Al-Gr and Al-Gr-ZrSiO4 hybrid composites](image)

There was a reduction of 10% (5.7 GPa) for Al – 1.3wt%Gr – 2.7wt%ZrSiO4 and 78% (1.4 GPa) reduction for Al – 2.6wt%Gr – 5.4wt%ZrSiO4 composite over unreinforced Aluminum with 6.3 GPa. This implies that deformation such as barrelling will be more pronounced in Al-Gr-ZrSiO4 composites than Al-Gr composites under similar application of compressive loading. The low compressive modulus in Al-Gr-ZrSiO4 might be due to the higher degree of segregation of Graphite particles in Al-Gr-ZrSiO4 composites as observed in Figures 4 and 5 compared to segregation in Al-Gr composites observed in Figures 2 and 3. High segregation of reinforcement in the matrix causes uneven distribution and is expected to degrade the elastic property of a material.

4. Conclusion

Al-Gr and Al-Gr-ZrSiO4 hybrid composites were produced by stir casting and their mechanical properties were investigated. Raw zircon and graphite were used without processing them and this resulted in an improvement in the strength characteristics of aluminum alloy. The results obtained indicated that the hybrid composite exhibited improved mechanical property consistently with increase in reinforcement. Aside from the impact strength and modulus of compression which decreases with increase in wt. % fraction of reinforcement in the hybrid composites, all other mechanical properties improved significantly. In contrast, the Al-Gr composites’ mechanical properties exhibited decrease with increase in weight fraction of the graphite reinforcement with exception of hardness which increase marginally. The results obtained suggests that the composites materials can be considered in applications such as pistons in light engine where required compressive strength is between 160 MPa and 200 MPa.

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Strength and Wear Characteristics of Aluminum/Zircon/Graphite Composites

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Abstract: The need to have materials with stable structural integrity has prompted the study of the strength and wear characteristics of hybrid composites of aluminium/zircon/graphite owing to the failure of monolithic alloys. In this study, varying amount (3, 7, and 12 wt. %) of zircon, graphite and the combination of the two (hybrid) particles were used as reinforcement in aluminium 6011 alloy. The cast samples were characterised for compressive strength, hardness, wear and morphological responses. For each reinforcement addition, composites' compressive strength ranges from 57.3 – 205.8 MPa while the hardness ranges between 44 – 140 HV in this study. Aluminium reinforced with the hybrid of zircon and graphite particles demonstrated superior properties over others. The morphology showed fine crystals of AlFeSi with increase in the concentration precipitated Mg 2Si phase in the aluminium matrix. Fluctuations in the wear characteristics of composites were observed except for Al/hybrid composite where wear volume rose steadily on application of 4.5, 8.9N and 13.35N for each filler content. All composites degrade at temperatures between 203-435°C, which implies that will maintain their properties in service within the temperature range. The composites will serve as potential materials for light automobile brake pads.

Keywords: Aluminum, Composite, Compressive strength, Graphite, Wear, Zircon

1. Introduction

Aluminum matrix composites (AlMCs) refer to the class of light weight high performance aluminum based material, usually 2xxx, 5xxx, 6xxx and 7xxx alloy series. These alloys are known for their effective fluidity, ease of casting, corrosion resistance and high strength-weight ratio (Singh et al., 2015). The properties of AlMCs can be tailored to the demands of different industrial applications via suitable combinations of matrix, reinforcement and processing route. These composites are being utilised in high-tech structural and functional applications in aerospace, defence, automotive, thermal management, sports and recreation due to their high strength to weight ratio (Surappa, 2003). Aluminum matrix can accommodate a variety of reinforcing agents including ceramics and organic wastes (in the form of fibres, whisker or particulates) with a maximum volume fraction of 70% (Surappa, 2003). Commonly used ceramic particles, which include silicon carbide (SiC), alumina (Al2O3) boron carbide (B4C), titanium carbide (TiC), graphite, quartz (SiO2) and zircon (ZrSiO4) have good hardness and wear characteristics, and these qualify them as potential reinforcing materials (Gopi et al., 2013; Raghavendra and Ramamurthy, 2014; Ramesh et al., 2009).

Zircon and graphite are chosen as reinforcement in this study because of their availability in the country. Graphite particle reinforced composites have applications in components requiring high wear resistance such as engine bearings, pistons, piston rings and cylinder liners (Barekar et al., 2008). Ramesh et al. (2009) observed that reinforcing Al6061 with graphite particles improves ductility, compressive strength and stiffness of the aluminum matrix but reduces its hardness. Macro and micro hardness of AA6082 Al alloy were found reduced on addition of graphite particles as reinforcement (Sharma et al., 2016). The density, ductility and tensile strength of the aluminum matrix with 0-12wt. % graphite (in a step of 3) reduce. Mechanical properties such as yield, tensile and compressive strengths of AA6082 have been enhanced by addition of Yttrium oxide (Y2O3) and...
graphite as reinforcements (Kumar et al., 2020) processed by friction stir casting. Zircon on the other hand is a by-product of cassiterite (tin ore) beneficiation (Bamalli, et al., 2011) and has been used as reinforcement in light metals especially where wear resistance with improved mechanical and thermal properties are required. It essentially consists of zirconium silicate (ZrSiO4) with hafnium, some rare-earth elements, titanium minerals and monazite, etc. It has also been used as ferro alloy and for facing of foundry moulds to increase the resistance against metal penetration (Rino et al., 2013).

Aluminum A356/ZrSiO4 MMC has been processed by Kumar et al. (2018) via stir casting. The reinforcement improves the wear characteristics of the composite and as a result, suitable for components such as cylinder sheets and cylinder blocks in automobiles. Zircon sand is a Neso silicate tetragonal crystal structured material with specific gravity between 4.60-4.71. Works on Al/zircon composite are enjoying increasing patronage as reinforcement in aluminum composites due to its unique properties (Kumar et al., 2015) such as its high refractoriness and resistance to sudden volume changes at elevated temperatures, resistance to abrasion, impact and chemical attack. The high hardness of Zircon (Moh’s hardness scale of 7.5) has made it a sought after potential reinforcement for aluminum (Kumar et al., 2015; Sucitharan et al., 2013). The impact of the combination of these two components on the morphology and mechanical properties of aluminum matrix needs to be appraised. This research is aimed at evaluating the mechanical and wear characteristics of aluminium 6011 alloy reinforced with zircon, graphite and the hybrid of these reinforcements.

2. Materials and Methods

2.1. Materials

The aluminium ingot (6011) was obtained from Tower Aluminium Rolling Mills, Ota, Ogun state, Nigeria. Ingot was cut with the use of a powered hack saw and further sheared into smaller pieces with the use of shear cutter. Graphite, zircon and magnesium, obtained from Peters Ventures, Lagos, Nigeria were milled to 150µm at Federal Institute of Industrial Research Oshodi (FIIRO), Lagos, Nigeria using a planetary ball Mill Machine (model 28A20 92). An electric furnace designed for the casting was built by assembling low density refractory bricks (light bricks) and electric resistant wire (Nichrome 80) as source of heat energy. The refractory bricks were obtained from Peters Ventures, Lagos, Nigeria while nichrome was obtained from Lightning Vapes, United States of America. The light bricks were 23cm x 7cm x 12cm in dimension, with a density of 0.763g/cm³. The Nichrome wire has a diameter of AWG 18 (0.102 cm), maximum operating temperature of 1180°C and resistance of 20 Ω.

2.2. Composite Production

The composites were produced by adding 3, 7 and 12wt. % of the filler (zircon and graphite) to aluminium matrix. To obtain the composition by weight for the hybrid (combination of zircon and graphite) reinforcement, the density ratio for each particle was used. Aluminum ingot was charged into the furnace while the reinforcement, wetting agent (magnesium) and mould were preheated to 300 °C. The reinforcement and the wetting agent were added to the molten aluminum at 750°C. The mix was stirred with a mild steel flat bar for 30 seconds and then poured into a mild steel metal mould of height of 135mm and diameter 15mm.

After pouring, the mould (and its contents) was allowed to cool to ambient temperature before removing the cast. The cast samples were solution heat treated at 530 °C for 3 hours in an electric resistant furnace and subsequently quenched in water. After quenching the samples, the samples were returned into the electric resistant furnace for artificial ageing at 180 °C for 5 hours.

2.3. Characterisations

2.3.1. Compression Test

The compression test was carried out on test samples using compression test equipment at the Centre for Energy Research and Development, Obafemi Awolowo University Ile-Ife. The samples were subjected to compression loads of 50 KN at 2 mms⁻¹. Compressive and fracture strengths were obtained from the test.

2.3.2. Hardness Test

The hardness test was conducted using a SIOMM Vickers hardness tester with model HVD – 10001S situated in the Metallurgical and Materials Department Laboratory, University of Lagos Nigeria. The samples were prepared by grinding them with emery paper of grit sizes 60, 150, 220, 320 and 400 in successive order to allow for proper microstructural view of the micro hardness tester indentation. The samples were indented for a dwell time of 10s and indentation was taken at three different points and the average results taken.

2.3.3. Scanning Electron Microscopy

The Scanning electron microscopy (SEM) test was carried out using a High-Performance Scanning Electron Microscope (VEGA 3 TESCAN) at accelerated voltage of 20KV to observe the microstructure of the samples, lines, pores and cracks at Department of Chemical, Metallurgical and Materials Engineering, Tshwane University of Technology, Pretoria, South Africa. The samples were prepared by grinding with emery papers with grit size 60, 150, 220, 320, 400, 600, 800 and 1200, in successive order. The samples to be observed under the SEM were mounted on a conductive carbon imprint left by the adhesive tape prepared by placing the samples on the circular holder and coated for 5 minutes to enable it conduct electricity.
2.2.4. X-Ray Diffraction
The X-ray diffraction (XRD) of the samples was conducted using PW1710 Philips X-ray diffractometer. Analysis was done with the use of PANalytical X’Pert High Score software for phase search-match in the Department of Chemical, Metallurgical and Materials Engineering, Tshwane University of Technology, Pretoria, South Africa. Samples were exposed to a monochromatic Cu Kα radiation (k = 1.5406 Å), operating at 40KV and 40mA. The samples were registered in a zero-background sample holder to avoid external background interferences. The diffractograms were registered in the range of 5° to 90° (2θ) in a step scan mode of 0.0170 at a scan step time of 1.00 second.

2.2.5. Wear Test
Wear rate measurements were performed using friction and wear testing equipment. To evaluate the durability of the materials in sliding contact with another surface under different conditions (load, distance, time and weight fraction) in this study, prepared samples were tested under dry sliding wear conditions. Dry sliding wear was carried out using a pin on disc equipment. Cylindrical specimens (both alloy and composites) of 15 mm diameter and 25 mm height were used as test samples. The specimen end surfaces were polished metallographically. Wear studies were conducted under varying conditions of load and sliding velocities. Measurement of wear loss of the pin was used to evaluate the volumetric loss (VL) during the wear test. Load was varied from 4.5, 8.9 and 13.35N was used to evaluate the volumetric loss (VL) during the sliding velocities. Measurement of wear loss of the pin were conducted under varying conditions of load and sliding wear test. The load was varied from 4.5, 8.9 and 13.35N while the disc speed was fixed at 123 rpm and time duration of 30s per wear session. A track radius of 7mm and varying sliding distance of 27, 54, 81 and 108 m were used for all the experiments. All the tests were conducted in air at room temperature. The weight loss method was used to calculate the wear rates. The wear rates of all the specimens were obtained. Furthermore, specific wear rate of the samples was calculated using the weight loss method given in Equation 1. The determination of the specific wear rate was based on derivations of Wear rate, Wear volume, Sliding distance and Weight loss. Formulas used are given in Equations (1)-(5).

\[ \text{Weight loss} = w_2 - w_1 \]  
\[ \text{Sliding Distance (m)} = 2\pi NDt \]  
\[ \Delta W = \text{Change in weight, } D = \text{disc diameter (100mm)} \]  
\[ \text{Wear Volume (m}^3) = \frac{\text{Weight loss (g)}}{\text{Density (g/cm}^3)} \times 1000 \]  
\[ \text{Wear rate (mm}^3/m) = \frac{\text{Wear Volume}}{\text{Sliding Distance}} \]  
\[ \text{Specific wear rate (mm}^3/Nm) = \frac{\text{Wear rate}}{\text{Load}} \]

2.2.6 Thermal Analysis
Thermal degradation of aluminium alloy and processed composites were determined with the use of Perkin Elmer thermogravimetric analyser (model TGA 17011909). Samples weighing 40mg were charged at 20 mL/min from 45°C to a final temperature of 850°C at a heating rate of 5°C/min. The heating was initiated with a residence time of 1min at 45°C before increasing the temperature. Temperature at the onset of degradation was recorded for each sample as a measure of composites, resistance to thermal degradation (thermal stability). This analysis was carried out at the Department of Chemical, Metallurgical and Materials Engineering, Tshwane University of Technology, Pretoria, South Africa.

3.0. Results and Discussion
3.1. Compression test
Figure 1 shows the compressive strength (CST) of aluminum reinforced with varying contents of particles (zircon, graphite and the hybrid). Composites’ CSTs are of higher magnitude than the unreinforced aluminum (0 wt. %) whose CST is 68.7 MPa, owing to improved strengthening effects of reinforcements on matrix. Aluminum reinforced with 3 and 7wt. % graphite was exceptions with strengthening effect of 57.3 and 29.9 MPa, respectively. Thus, the use of hybrid reinforcement will enhance the CST of aluminum as it displayed superlative results compared to other singly/monolithic used reinforcements. Aluminum’s CST increased from 68.7 MPa to 114.4 and 305.8 MPa with the use of 3 and 7wt. % hybrid reinforcements respectively. The results here are superior to that obtained with the use of dolomite and calcium carbonate as separate fillers in reinforcing phenolic resin mixed with graphite and alumina to design brake pads carried out by Ruzaidi et al. (2013), where CST of approximately 80 and 90MPa for calcium carbonate and dolomite fillers respectively was observed. Trends of samples’ CST are similar to their fracture strengths with approximately same magnitudes as shown in Figure 2. This implies that most of the samples fracture at their maximum compressive stress values; hence, material with the highest magnitude of CST will be of importance during selection. Incorporation of 3, 7 and 12 wt. % zircon equally improve aluminum’s fracture strength.

Results in this study have shown that the fracture strength of aluminum is improved at any composition of the zircon/graphite hybrid filler with 7wt. % hybrid filler having a superlative resistance to fracture under compression as it fractures at 206.8MPa. At the same filler content, Al/3wt.% zircon possesses fracture strength of 106.4MPa, which is the best achieved for Al/zircon composite while Al/graphite composite exhibits a maximum fracture stress of 99.5MPa at 12wt. % graphite addition. Comparing samples with similar particle content, Figure 2 shows that hybrid of zircon/graphite impacts the best fracture stress.
When compared with the control sample (0 wt. % filler content with 95HV), it is observed that reinforcing 6011 aluminum yields improved hardness at some filler contents. Addition of 3, 7 and 12wt. % zircon to aluminum yields 108, 103 and 66HV, respectively. The least hardness value of 45HV, which is ≈53% lower than the control specimen (95HV), is attained at 3wt. % graphite while 58 and 135HV are obtained in composite with 7 and 12wt. % graphite, respectively. Reinforcement with 12wt. % hybrid produces the highest magnitude of 140HV.

3.3. Morphology

The morphologies of unreinforced aluminium and its composites as captured by SEM are discussed in Figures 4-12. The morphology of as-cast 6011 aluminum (see Figure 4a) shows the presence of dark black crystals of Mg2Si phase in the alloy distributed over the α–aluminium matrix. The Mg2Si intermetallic has been confirmed to provide strength and hardness when finely dispersed in the alloy (Velasco et al., 1995).

According to Qin et al. (2007), an intermetallic compound of Mg2Si contributes to good mechanical properties of alloy by providing high hardness, reasonably
high elastic modulus and low thermal expansion. The morphology also reveals the existence of Fe-rich intermetallic compound, which has been identified as AlFeSi. In the as-cast sample shown in Figure 4a, this phase occurs as tiny crystals scattered over the surface of the matrix. This fine Fe-rich intermetallic phase was discovered by Shouxun et al. (2013) to maintain a chemical structure of "α" Al8Fe2Si. This compound is regarded as the most undesired because it causes brittleness (Mohammad et al., 2016). Salleh et al. (2015) agree that the effect of iron in aluminium alloys can lead to reduced tensile strength and ductility. The phases present in this material are comparable to that observed in as-cast aluminium processed by Adeosun et al. (2016a and b). The XRD pattern of the unreinforced aluminium alloy in Figure 4b shows the presence of intermetallic phases of AlFeSi and Mg2Si. Aluminum matrix exists on 2θ = 37.4, 44, 64.4 and 78° (Kumar et al., 2018). Diffractions on 2θ = 46 and 58.9° represent the presence of AlFeSi and Mg2Si respectively (Farshidi et al., 2018; Kumar et al., 2012; Wunderlich et al., 2014; Kumar et al., 2016a).

Micrograph of aluminium reinforced with 3 wt.% of zircon at low (x200) and high (x600) magnifications is shown in Figure 5a and b. The image shows a uniform distribution of reinforcement (white spots) in the aluminium matrix at higher magnifications (see Figure 5b). At a lower magnification (see Figure 5a), the Mg2Si intermetallic phases have significantly reduced geometry while the AlFeSi phase is of a lower concentration with few clusters of its crystals. Observing the microstructure at a higher magnification, zircon particles are shown to be strongly bonded to the matrix as both Mg2Si and AlFeSi precipitates are embedded in the microstructure. Combined influence of well bonded zircon particles with existence of Mg2Si could be responsible for the composite’s improved resistance to compression and indentation when its hardness and compression values are compared with that of the unreinforced sample.

The existence of zircon in the alloy is generally affirmed to increase the tensile strength of aluminium but increase in its content as reinforcement will evidently enhance brittleness thereby lowering its elongation (Rammath et al., 2018). Zircon in the composite is evidenced by the diffraction on 2θ = 27 and 36° (Figure 5c). These are some of the diffractions of zircon observed by kumar et al., (2018) where A356 aluminium was reinforced with zircon. X-Ray diffraction of zircon particles characterised by Genoveva et al., (2007) shows peaks at 2θ = 27.08 and 35.69° Figure 6a is the micrograph of aluminium reinforced with 7 wt. % zircon at x200, which shows denser Mg2Si phase (compared to 3 wt. % zircon). This phase is found to elongate along the deformation direction with few crystals precipitating on the slip lines as shown at higher magnifications of x200 and x600 (Figure 6a and b). These tend to impede dislocations, thereby creating improved deformation resistance of sample. The AlFeSi phase shows reduced concentration in the matrix while the zircon and Mg2Si particles are uniformly distributed on the aluminium matrix.

Increasing the volume of reinforcement to 12wt. % (see Figure 7), poor bonding of Al/zircon is revealed in the microstructure at higher magnification with evidence of visible cracks distributed over the grey aluminium matrix. This is accompanied with reduced Mg2Si volume fraction with increase in AlFeSi phase, which is responsible for the reduction in compressive strength and fracture stress.
Reinforcing aluminium with graphite of a similar content (3 wt. %), there exists clusters of the reinforcement (see Figure 8), which appears to form on the surface making the Mg₂Si phase look invisible compared to Al/zircon composite shown in Figures 5-7. There are very few Mg₂Si crystals on the surface of the composite with Fe-rich intermetallic crystals, which clustered at a point as in Figure 7b (higher magnification). This is responsible for the cause of decrease in composite’s, compressive strength, fracture strength and hardness (see Figures 2 and 3) compared to Al/zircon of the same filler content. The morphology of the Al/graphite composite also gives reasons for the diminished mechanical properties compared to unreinforced aluminum (see Figure 4a), which reveals volume fraction of Mg₂Si phases.

Figure 8. SEM (a) x200 (b) x600 and (c) XRD of Al/graphite (3wt. %) composites

The graphite particle in the composite is diffracted at 2θ = 26.3° (see Figure 8c) and this is comparable to the findings of Peng et al., (2013) and Sayah et al., (2018) where graphite’s XRD analysis shows diffraction at 2θ = 26.5°.

The morphological effect of reinforcing aluminum with 7wt. % is shown in Figure 9. Few particles of AlFeSi, are observed to be fairly dispersed in the microstructure with Mg₂Si intermetallic crystals clustered at certain regions. The morphology of the α-Al matrix appears rough with few cracks caused by the presence of un-bonded graphite. The lack of proper bonding between graphite and matrix may be the reason for the least compressive and fracture strengths observed in Figures 1 and 2.

The morphology of aluminum reinforced with 7wt. % graphite (see Figure 10a) shows the presence of few Mg₂Si scattered on the surface of the composite. The presence of graphite and Fe-rich intermetallic (AlFeSi) is also seen on the matrix surface. Figure 10b shows a higher magnification. The microstructure indicates good interfacial bonding between graphite and α-aluminum matrix. The maximum increase in hardness of this composite (see Figure 3) is attributed to good interfacial bonding between matrix and reinforcement and the presence of the hard Fe-rich crystals. The AlFeSi intermetallic crystals are formed along the visible stress direction (which could have occurred during solidification) while the Mg₂Si phases are not formed on these sites. At maximum graphite content of (12wt. %) the flow of Mg₂Si crystals along solidification direction could be prevented. Existence of more AlFeSi crystals compared to that of Mg₂Si may be responsible for this occurrence.

Figure 9. SEM of Al/graphite (7wt. %) composites (a) x200 (b) x600 (c) x1000

Figure 10. SEM of alumina/graphite (12wt. %) composites (a) x200 (b) x600

Very fine AlFeSi crystals are produced in 3wt. % hybrid composite shown in Figure 11 and a decline in volume fraction is observed in the matrix than those observed in previous morphologies. The microstructure shows considerable amount of Mg₂Si sparsely dispersed in the aluminum matrix with few clustering sites. A higher magnification of the microstructure (see Figure 11b) shows clearly the clusters of Mg₂Si with clusters of both Mg₂Si and graphite phases, resulting into a thick layer on the surface coupled with the existence of fine crystals of zircon. It is observed that the compressive and fracture
strengths of this composite are superior to both aluminum/graphite and aluminum/zircon composites for each content of reinforcement addition. This implies that these clusters have strong interface with the matrix. Addition of 12wt. % graphite/zircon (see Figure 12) enhances more precipitations of Mg$_2$Si phases with formation of thicker Mg$_2$Si/graphite/zircon layer. X-Ray Diffraction of Al/Hybrid composite in Figure 12c shows the presence of graphite and zircon coupled with the intermetallic phases.

Figure 11. SEM of hybrid (3wt. %) composites (a) x200 (b) x600

Figure 12. SEM (a) x200 (b) x600 of hybrid (12wt. %) composites (c) XRD of hybrid (3wt. %) composites

3.4. Wear Test

3.4.1. Wear volume

Wear volume describes the amount of worn debris occurring during dry sliding wear and is fundamental to explaining the tribological behaviour of materials. Figure 13a shows the wear volume in mm$^3$ of Al/Zircon composites at loads of 4.45N, 8.9N and 13.35N. There is a general reduction of wear volume compared to 3wt. % Al/Zircon composites except at 7wt. % where wear volume increases for 13.85N. The reduction is attributed to increased hardness (as there is an increase in volume fraction of zircon particles in the matrix) but does not vary significantly with increasing weight fractions between 7 and 12 wt. %. It can also be observed that for 7 and 12wt. %, wear volume increases with increase in loads except for composites at 3wt. %. The exemption might be due to clogs of debris preventing contact between the disk and pin during wear as increase in load is expected to lead to increased wear volume.

Figure 13. Wear volume of composites (a) Al/zircon (b) Al/Graphite (c) Al/hybrid
wear volume. Wear volume reaches peak for each of the loads at 7wt. % graphite. The initial increase in wear can be attributed to reduced fracture toughness of graphite. However, as the graphite increases in weight fraction, presence or formation of thick solid lubricant film, which overrides the effect of fracture toughness, is responsible for the reduced wear volume (Ted and Chi, 2000). It can also be observed that the range of wear volume of Al/Graphite is less than in Al/zircon composites due to the resistance to wear shown by graphite via forming lubricating films on the surface during wear. The wear volume of hybrid composites at 4.5, 8.9 and 13.45N are shown in Figure 13c. It is observed that increase in load leads to increase in wear volume at all weight fractions. Wear volume with similar pattern as Al/graphite composites, increases up to 7wt. % before declining. This suggests that presence of graphite plays a significant role in the volume of wear than zircon particles.

### 3.4.2. Specific Wear Rate

Figure 14a-c shows the specific wear rate of aluminum composites at 4.45N. For Al/zircon composites, specific wear rate reduces after 3wt. % compared to others at higher weight fractions. The increase in specific wear rate can be attributed to increase in hardness of the composites as hard zircon particles are expected to heighten the hardness of the composites. Al/Graphite and Hybrid composites show the highest specific wear at 7wt. % before subsequently declining. The behaviour of Hybrid composites is attributed to the contributions of the reinforcements to the resistance of wear. The hard reinforcement (zircon) is expected to protect the matrix from wearing off while the soft reinforcement (graphite) is expected to form a tribo layer to resist wear. Thus, there is expected to be improved wear reduction. However, the Figure shows higher specific wear at 7wt. % compared to Al/Graphite and Al/Zircon composites. Figure 14b shows the specific wear of aluminum composites at 8.9N. Al/Zircon composites show similar pattern in behaviour to what is observed in Figure 5a. The specific wear rates of Al/Graphite and hybrid composites increase up to 7wt. % before declining.

**Figure 14.** Specific wear rates of aluminium composites (a) 4.5N (b) 8.9N (c) 13.35N

Figure 14c shows the specific wear of aluminum composites at 13.35N. Al/Zircon composites show reduction in specific wear rate with increasing weight fractions.

### 3.5. Morphology of worn samples

The SEM images of worn surfaces of unreinforced aluminum and its composites in Figure 15. Surface debris, groves and cracks are observed on samples after wear indicating delamination and abrasion (Idusuyi and Olayinka, 2019; Kumar et al., 2016b; Kumar et al., 2018; Kumar et al., 2020). Shallow groves and minor cracks with direction of metal flow are observed on the worn surface of unreinforced aluminum (see Figure 15a) while severe cracks with deep groves are observed on Al/Zircon worn surface (see Figure 15b). This form of crack, according to Rajesh et al., (2018) grows and culminates in long thin wear sheets after shearing to the metal surface.

More pits are formed on the worn surface of Al/Graphite (see Figure 15c) compared to worn surfaces of unreinforced Al and Al/Zircon. The pits elongate along the direction of deformation which makes them look parallel along metal flow orientation. Ghosh et al. (2012) attribute this to micro cutting tendency which further explains abrasion/abrasive wear. There exist groves, pits
and cracks with some thick debris on the worn surface of Al/Hybrid composite (see Figure 15d).

Figure 15. SEM of worn samples (a) unreinforced aluminum (b) Al/Zircon (c) Al/Graphite (d) Al/Hybrid composites x600

3.6. Thermal Stability
Determination of thermal stability of composites is highly required in wear applications. This is due to the heat generated during friction, which can distort the properties of materials being used. Figure 16 shows the onset of degradation temperatures of unreinforced aluminum and its composites via reinforcement with zircon, graphite and the hybrid.

Figure 16. Onset decomposition temperatures of unreinforced aluminum, Al/zircon, Al/Graphite and Al/Hybrid composites

Degradation temperature of Al/Zircon composites increases with filler contents from 203- 280°C. Graphite-reinforced aluminum samples show no significant change in thermal degradation profile as a constant temperature of 400°C is maintained for 5, 7 and 12wt. % reinforcements. The maximum temperature that can be attained before the hybrid composites start deteriorating in properties is observed at 435°C 7wt. % additions while the least temperature it can withstand is 304°C at 5wt. %. All composites will display good performance between 203-435°C, which is greater than that observed for unreinforced aluminium which starts deteriorating at 197°C.

4. Conclusion
This study investigated the wear and mechanical characteristics of Al/Graphite, Al/Zircon and A/I/Zircon/Graphite (Al/Hybrid) composites. For each category of filler addition, hardness, compressive and fracture strengths are of the highest magnitudes when the hybrid of zircon and graphite reinforcements is used above 3wt. %.

Beyond this filler content, precipitation of Mg2Si is much pronounced within the Al matrix. Wear volume of Al/Hybrid composites increases with increasing load compared to Al/Graphite and Al/Zircon composites. There are fluctuations in the wear characteristics of composites except for Al/hybrid where wear volume rose steadily on application of 4.5, 8.9N and 13.35N for each filler content. The composites will serve as potential materials for light automobile brake pads.

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References:
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Thermal Performance of Different Shapes of Solar-powered Oven for Meat Processing and Preservation

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Abstract: A solar oven is a device used to generate heat energy through the radiation of solar energy in a confined space. It is widely used especially in food processing and preservation with the box-shape solar oven being the common geometry. In this study, the thermal performance of different geometries (block-type and curve-type) of the solar oven and the drying of meat samples in the device are investigated. The solar oven designs were analysed numerically using a computational multiphysics software for fluid and heat transfer after it had been validated. Results obtained shows that the curve-type box solar oven with the meat samples well-spaced had the better thermal performance. Hence, the curve-type box model was adopted and used in fabrication for experimental analysis. The numerical results were compared to the results from the experiment conducted, and the outcome of this comparison showed that the curve-type solar oven design with the meat samples largely spaced ensured the drying effect. The meat samples for the optimal design dried uniformly after 40 minutes and the average percentage difference between the numerical and experimental results was approximately 2.75%. The findings would shed lights in improving the effectiveness and processing time of existing box solar ovens.

Keywords: Solar oven, Thermal performance, Block-type, Curved-type, Heat transfer, Food processing

1. Introduction

A large number of the Nigerian population in relation to other developing nations in Africa, Asia, and Latin America, rely on energy resources such as biomass and firewood for cooking, baking, and boiling of water. Recent studies (e.g., Fernandez et al. (2002); Bala et al. (2002); Garba and Bashir (2002); Bello et al. (2016)) show that these heat utilisation activities are liable for over 70% of the energy needs of most homes. The complete reliance on limited petroleum products like liquefied petroleum gas otherwise called natural gas, kerosene and electricity for everyday energy needs is of great concern. There is major need for steps to be taken to protect the earth from environmental changes brought about by acid rain, global warming and other ecological degradations, which have unfavourable outcomes on the earth (Yusuf et al., 2014).

In the 1970’s, Barbara Kerr and Sherry Cole, two of the founders of Solar Cookers International initiated the concept of the solar cooking box-type oven. Their design which consist of cardboard and foil, is a model of simplicity and efficiency.

The usage of solar ovens is not restricted to food preparation, but is the foundation for several industrial and energy applications. Technically, a solar cooker is a solar thermal collector designed precisely for heating solids such as food (although it can also be used for liquids kept in containers). The determining parameter of cooked foods is the temperature; hence, the design of a solar cooker is focused towards achieving a rapid increase of the temperature for the food.

Temperature increase in a solar kitchen can be attained by trapping the maximum possible solar radiation, in other words, accumulating the heat energy more in an insulated box. The concept of heat concentration in solar ovens can be observed in various real cases like automobiles, residences and the atmosphere. The accumulation of heat is achieved through the enclosure of transparent windows (Diz-Bugarín and Rodriguez-Paz, 2011).

Solar energy is a major source of energy considering the search for alternatives of domestic fuel. It is one of the purest energy sources which is free from environmental hazards and it is readily available and infinite. However, like the development of all other energy sources, the revolution of solar energy into the technological world will require a lot of planning, organisation, generation and dissemination of information alongside providing of infrastructures to harness, it is an efficient and effective means. The total solar power that is incident on the surface of the earth from the sun is $1.5 \times 10^{18}$ kWh annually, which is equivalent to $1.9 \times 10^{14}$ ton coal equivalent (Tec). Sunita and Prabha (2009) argued that while compared to the annual world consumption of almost $10^{16}$ Tec, it is a large amount and approximately 10,000 times more than what is consumed on the earth annually.

Due to the need for food to be preserved, the existing demand of high-quality foods in the food market involves
dried products with high nutritional and organoleptic properties with similar levels as found in fresh products. Drying of food products is very crucial because it is the easiest and the most familiar means of preserving food. Drying prolongs the shelf life of the food. The extraction of surplus water from vegetables and other food products by the conventional technique of open sun drying is not efficient enough, since the products become infected with bacteria and insects and decay rapidly in the high ambient temperatures and relative humidity (Ayensu, 1997; Ratti and Mujumdar, 1997).

The box solar oven is a device used for drying food with use of the Sun (solar energy) while the food is kept within a confined space. In the course of this work, designs of varying geometries are analysed and compared with one another. These designs were developed based on the past jobs stated below as modifications were implemented to improve its performance.

Based on past works, a solar dryer was designed and constructed for the drying of green peas while its drying performance was compared with spreading the green peas directly under the sun. The outcome of his exercise showed that the drying rate of the solar dryer on the green peas was found to be higher than that of the open sun-dried peas (Sunil and Sharma, 2013). A solar oven was also constructed using reflectors while another was constructed without a reflector. Their performances were analysed and it was inferred that the box type solar oven with reflectors is 99% efficient compared to that without reflectors which is just 96% efficient (Yusuf et al., 2014). Another case is that of a box solar oven constructed with and without a wiper mechanism. The comparison of both shows that due to the accumulation of water vapor on the surface of the glass lid where the entrant of the sunrays takes place, the solar oven without the wiper mechanism was more efficient than that with the wiper mechanism (Ademe and Hameer, 2018). The adoption of the box solar oven would lead to enhanced processing of meat at reduced processing time and cost while preserving the natural quality of the meat. The box solar oven could be used conveniently in remote areas where conventional power supply is a major challenge.

2. Numerical Model and Procedure

The numerical investigation was conducted by first obtaining a geometric model for both solar oven designs. Details of the dimensions of the oven and meat samples are described below. To assess the drying effect on the meat samples, the meat samples were modeled as cubic shapes of length 30 mm.

2.1 Model description

The block-type and curve-type solar oven models were analysed using materials and features shown in Figure 1. The external structure is made of aluminium due to its high thermal conductivity of 202.1 Wm⁻¹K⁻¹ and its availability. For purpose of preventing heat loss, wood which has thermal conductivity of 0.173 Wm⁻¹K⁻¹ was used for lagging. A glass lid was used at the top of the oven for ease of penetration of solar energy into the oven and to ensure that the heat gained from the solar radiation is conserved. The glass has a thermal conductivity of 1.5 Wm⁻¹K⁻¹. Two vents are placed at each of the oven side walls to aid escape of moisture from the meat. The vents also ensure that moisture does not accumulate on the inner surface of the glass. Sixteen cube-shaped meat samples were placed on a mesh platform of 10 cm high in order to ensure that the heat within the oven is transferred to all meat samples.

![Physical Model of Box Solar Oven](image1)

Figure 1. Physical Model of Box Solar Oven

Figure 2 shows the geometric model of the block-shaped and curved-type solar ovens while Figure 3 shows the geometry of one meat sample. In table 1, all the dimensions of the solar-oven and meat samples are shown.

![Dimensions of Different Designs of Solar Oven](image2)

Figure 2. Dimensions of Different Designs of Solar Oven - (a) Block-type Design and (b) Curved-type Design
Figure 3. Dimensions of Meat Sample

Table 1. Dimensions of Physical Model of Box Solar Oven and Meat Sample

<table>
<thead>
<tr>
<th>Nomenclature</th>
<th>a</th>
<th>b</th>
<th>c</th>
<th>d</th>
<th>e</th>
<th>f</th>
<th>g</th>
<th>h</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dimensions</td>
<td>300</td>
<td>700</td>
<td>450</td>
<td>500</td>
<td>450</td>
<td>20</td>
<td>500</td>
<td>30</td>
</tr>
</tbody>
</table>

Figure 4 shows the meat samples with small and large spacing. The samples are also numbered to track the temperature of each sample within the oven. The large and minimum spacing dimensions are highlighted in Table 2 and 3, respectively.

Table 2. Dimensions of Meat Positions for Large Spaced Meat Samples

<table>
<thead>
<tr>
<th>Nomenclature</th>
<th>Dimensions (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>i</td>
<td>140</td>
</tr>
<tr>
<td>j</td>
<td>60</td>
</tr>
<tr>
<td>k</td>
<td>110</td>
</tr>
<tr>
<td>l</td>
<td>100</td>
</tr>
<tr>
<td>m</td>
<td>50</td>
</tr>
</tbody>
</table>

Table 3. Dimensions of Meat Positions for Minimum Spaced Meat Samples

<table>
<thead>
<tr>
<th>Nomenclature</th>
<th>Dimensions (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>n</td>
<td>175</td>
</tr>
<tr>
<td>o</td>
<td>200</td>
</tr>
<tr>
<td>p</td>
<td>350</td>
</tr>
<tr>
<td>q</td>
<td>10</td>
</tr>
</tbody>
</table>

There were four cases of oven design and meat spacing considered in this study. These are:
1) Block type solar oven with minimum meat cut spacing (BMCS).
2) Block type solar oven with large meat cut spacing (BLCS).
3) Curved type solar oven with minimum meat cut spacing (CMCS).
4) Curved type solar oven with large meat cut spacing (CLCS).

All four scenarios were numerically simulated under similar conditions. Figures 5 shows the geometric model of the BMCS, BLCS, CMCS and CLCS models.

Figure 4. Numbering and Spacing of Meat Samples: (a) Large Spacing and (b) Small Spacing

Figure 5. Different Shapes of Solar Oven with Meat Sample Spacing: (a) BMCS (b) BLCS (c) CMCS, and (d) CLCS
2.2 Validation of Computational Code

A computational multiphysics software for fluid and heat transfer was used to generate numerical results for the drying of the meat samples in the solar oven. The numerical results were validated by comparing with the experimental results obtained from the collector outlet air temperature (Sunil et al., 2013). Their work was on modelling the drying kinetics of green peas in a solar dryer under open sun. The physical model of the experiment is shown in Figure 6.

![Figure 6. Experimental Set-up](source: Based on Sunil et al. (2013))

The experiment set up consisted of an indirect natural convection solar dryer with a flat-plate solar air collector, a drying chamber and two drying trays. The details of the solar collector assembly and drying chamber are shown in Tables 4 and 5, respectively.

![Table 4. Details of Solar Collector Assembly](source:)

<table>
<thead>
<tr>
<th>Parameters</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>a Collector Area ($A_c$)</td>
<td>0.60 m$^2$</td>
</tr>
<tr>
<td>b Length</td>
<td>1.2 m</td>
</tr>
<tr>
<td>c Width</td>
<td>0.5 m</td>
</tr>
<tr>
<td>d Absorber plate</td>
<td>0.5 mm aluminum sheet</td>
</tr>
<tr>
<td>e Surface treatment</td>
<td>Black paint coating</td>
</tr>
<tr>
<td>f Glazing</td>
<td>Normal window glass of thickness 4mm</td>
</tr>
<tr>
<td>g No. of glazing</td>
<td>Two</td>
</tr>
<tr>
<td>h Back insulation</td>
<td>Polystrene of thickness 4cm</td>
</tr>
<tr>
<td>i Casing</td>
<td>Made of plywood</td>
</tr>
<tr>
<td>j Collector slope</td>
<td>32° (latitude)</td>
</tr>
<tr>
<td>k Airflow area (at inlet and outlet)</td>
<td>0.02 m$^2$ and 0.0038 m$^2$</td>
</tr>
<tr>
<td>l Distance between glazing</td>
<td>10 mm</td>
</tr>
<tr>
<td>m Distance between cover and absorber plate</td>
<td>40 mm</td>
</tr>
</tbody>
</table>

Figure 7 shows a comparison between the experimental results in Sunil et al.’s work and the numerical results obtained using the computational software used in the present study. The plot shows a similar trend with an average difference in temperature of 8.12%. This shows that the chosen computational tool gives good results and can be used in this present study.

![Figure 7. Comparison of Experimental Results and Numerical Results for Collector Outlet Air Temperature](source:)

2.3 Governing Equations and Boundary Conditions

Equations (1) to (3) are the governing partial differential equations for mass, momentum and energy for flow and heat transfer within the oven. The boundary conditions applied to the solar oven in this study are as follows:

It was assumed that the heat flux was constant at the glass surface as a result of the location when the experiments were to be carried out and the oven surface area.

1) No internal heat was generated within the solar oven, and
2) Negligible heat loss was assumed at external walls.

The numerical simulations were carried out for three-dimensional transient convection and radiative heat transfer.

\[
\frac{\partial \rho}{\partial t} + \nabla \cdot (\rho \mathbf{v}) = 0 \tag{1}
\]

\[
\frac{\partial (\rho \mathbf{v})}{\partial t} + \nabla \cdot (\rho \mathbf{v} \mathbf{v}) = -\nabla p + \rho \mathbf{g} + \mathbf{F} \tag{2}
\]

\[
\frac{\partial (\rho h)}{\partial t} + \nabla \cdot (\mathbf{v} \rho h) = \nabla \cdot (k \nabla T) + S_h \tag{3}
\]
2.4 Numerical Results and Discussion

The numerical simulation is carried out for meat samples heated within the oven for 2 hours in intervals of 20 minutes. The meat samples are assumed to be fully dried at a temperature of between 60°C to 70°C. The results for the four cases considered in this study are discussed in this section. Figure 8 shows the temperature results for each meat sample over a period of 120 minutes for the BMCS design.

The temperature values of each meat sample were recorded in intervals of 20 minutes. Based on the range of temperature for fully dried meet samples, the results presented show that for the BMCS design, all the meat samples were fully dried at 60 minutes. Meat sample 16 had the highest temperature of 82.77°C while meat sample 14 had the lowest temperature of 64.54°C.

Figure 9 shows the temperature results for each meat sample over a period of 120 minutes for the BLCS design. The temperature values of each meat samples were recorded in the BLCS solar oven in intervals of 20 minutes. The results obtained for this design show that all the meat samples were fully dried at 60 minutes. Meat sample 10 has the highest temperature of 80.56°C while meat sample 9 has the lowest temperature of 61.23°C at 60 minutes.

The numerical results for the CMCS solar oven design are presented in Figure 10. All the meat samples were dried at 60 minutes but were at higher temperatures than when placed in the block-type solar oven. Results showed that meat sample 8 recorded the highest temperature of 103.20°C, while meat sample 5 has resulted the lowest temperature of 70.09°C at 60 minutes. The numerical results for the CLCS solar oven design are shown in Figure 12. The meat samples were uniformly dried at 40 minutes with lowest temperature of 62.58°C for meat sample 11 to highest temperature of 67.50°C for meat sample 16. The CLCS was the chosen design for experimental testing because it dried the meat samples uniformly at a shorter time of 40 minutes.
3. Experimental Model and Procedure

3.1 Model Description

The fabrication of the chosen curved-type solar and results obtained from the experimental tests are presented in this section. The materials selected and their sizes are, 1.2m by 2.4 m by 0.0007m aluminum sheet, 5 mm thick plywood and 3 mm thick glass lid. Figure 12 shows the picture of the un-insulated curved-type solar oven fabricated for the experimental tests.

![Figure 12. Curve-type Solar Oven without Insulation](image1)

The next phase of fabrication was the insulation of the aluminum frame using the 5 mm thick plywood cut into various shapes that fit on the external walls of the oven. The walls of the oven were perforated at the side walls as the vents. Afterwards, a rectangular mesh of height 10 cm was constructed in order to serve as a platform upon which the meat samples are to be dried are placed as shown in Figure 13.

![Figure 13. Fully Fabricated Curve-type Solar Oven](image2)

A digital thermometer was used in measuring the temperatures of each meat sample in intervals of 20 minutes. The picture of the thermometer and its specifications are shown in Table 6. The thermometer uses infra-red technology to obtain the temperature of the meat samples.

![Table 6. Specification of digital thermometer](image3)

<table>
<thead>
<tr>
<th>TS600 Model</th>
<th>Features</th>
<th>Specifications</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Temperature Range</td>
<td>-50°C to 600°C (58°F to 1112°F)</td>
</tr>
<tr>
<td></td>
<td>Accuracy</td>
<td>+/- 1.5% or +/- 1.5°C</td>
</tr>
<tr>
<td></td>
<td>Emissivity</td>
<td>0.01 – 1</td>
</tr>
<tr>
<td></td>
<td>Reflectivity</td>
<td>0.95</td>
</tr>
</tbody>
</table>

3.2 Experimental Procedure

The sun which is our major source of energy to power this curve-type box solar oven is known to rise from the East and set at the West within 12 hours of the day. The oven was placed in accordance to the orientation below relative to the trajectory path of the sun as shown in Figure 14. The glass lid is opened to allow for accurate temperature measurement of the meat samples every 20 minutes without interference from the glass lid. The meat samples were placed in the oven. The experiments were carried out in November 2019 and the first temperature measurement taken at 11:00am.

![Figure 14. A Descriptive View of the Solar Oven’s Positioning Relative to the Trajectory of the Sun](image4)

3.3 Comparison between Experimental and Numerical Results

Figure 15 shows the temperature of the meat samples recorded during the period the experiment was conducted. The experimental results show that the meat samples were uniformly dried at 40 minutes which is the same time achieved numerically. The lowest temperature obtained experimentally is 62.8°C for meat sample 16 and the highest temperature of 68.4°C for meat sample 4. Figure 16 shows the comparison between the numerical and experimental results obtained for the CLCS solar oven design.

The results at 40 minutes were tabulated and compared as shown in Table 7. The average percentage difference between the experimental and numerical temperature
results of the meat samples at 40 minutes drying time is approximately 2.75%.

4. Conclusion

It was discovered that the curve-type design is more efficient than the block-type design as there was improved molecular movement due to the reduced volumetric space implemented. This aided the quicker generation of solar heat energy within the oven. Also, the position of the meat samples is a very important factor as it is observed that the cases where the meat samples had minimum spacing, the temperatures were poor compared to cases in which the meat samples had large spacings. This improvement due to positioning was due to the radiation of the heat energy absorbed by the aluminum sheet which has quite a high thermal conductivity.

The effect of convection also took place through the vents created as it helped the escape of moisture instead of having it accumulated on the surface of the glass lid internally, thereby, causing poor effects of the solar heat flux. This outcome brings about improvements in the drying effect of existing designs of box solar ovens. Time which a very vital parameter being considered even in the use of this device is saved.

References:


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Tolulope Theophilus Areo is presently a practicing Automobile Engineer in Lagos, Nigeria. He completed his Bachelor of Science Mechanical Engineering, at the University of Lagos, Akoka, Lagos, in 2010. His current research interest is in the area of harnessing solar energy for the storage of food.

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Study of Effects of Afraegle Paniculata Extract on Corrosion of Galvanised Steel in H₂SO₄ Solution

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Abstract: The study investigated the corrosion inhibition properties of Afraegle Paniculata extract in 1 M H₂SO₄ solution on galvanised steel with a view to understanding the reaction mechanisms as well as kinetics and thermodynamics of adsorption step in the corrosion reaction. The experimental investigation was conducted using weight-loss and gasometric techniques. The surface morphology of the exposed steel was studied using scanning electron microscope (SEM) and energy dispersive X-ray spectroscopy (EDS). The results of this study revealed that the acidic medium increased the corrosion rate of galvanised steel and the corrosion reaction followed a first order reaction kinetics with a rate constant (k) of 23.7 mm day⁻¹ M⁻¹. It was deduced that the adsorption of A. Paniculata extract on galvanised steel surface obeyed the Langmuir adsorption isotherm with both reaction rate and Standard Change in Gibbs Free Energy of adsorption (ΔG°ₚₖₛₖ) increasing with increase in temperature. Different values of Standard Change in Gibbs Free Energy of adsorption (ΔG°ₚₖₛₖ) at different temperatures showed that the reaction was thermodynamically feasible and spontaneous. The Standard Change in Heat of Reaction (ΔH°ₚₖₛₖ) and Standard Entropy Change (ΔS°ₚₖₛₖ) were found to be -10.88 kJ mol⁻¹ and -0.139 kJ mol⁻¹ K⁻¹, respectively. The SEM images confirmed that galvanised steel corroded in acidic environment and A. Paniculata extract inhibited the rate of corrosion in galvanised steel in 1 M H₂SO₄ solution. The SEM images revealed that cracks on the metal surface enhanced corrosion, and the corrosion rate were directly proportional to both the number and sizes of cracks. Weight loss, SEM and EDS analyses revealed that galvanised steel can corrode in 1 M H₂SO₄ and that corrosion rate could be reduced by using A. Paniculata extract as an inhibitor.

Keywords: Afraegle Paniculata, Corrosion, Weight Loss, Gasometric, Inhibition, Adsorption

1. Introduction
The transportation of crude petroleum oil and gas in steel pipelines faces several challenges including corrosion due to entrained carbon dioxide and/or hydrogen sulphide (Bai and Bai, 2005). Corrosion causes the degradation of metals due to the transfer of electrons between the solid metal surface and the acidic solution formed as a result of the dissolution of carbon dioxide and/or hydrogen sulphide in water (Ogunrinde and Aribike, 2019; Abdel-Gaber, 2009; Abiola and James, 2009). Corrosion has been identified as one of the major causes of damage and failure in petroleum oil and gas pipelines. The transmission of petroleum crude oil and gas entrained with carbon dioxide, hydrogen sulphide (H₂S) and water in steel pipelines can cause severe corrosion problems such as reduction in strength and life-span of steel and transmission pipes, loss of metallic properties, wastage of metal, production losses and open cracks in the pipes. Thus, several experimental studies have been conducted in recent past to investigate prevention of corrosion in steel metals (Ma et al., 2000; Tang, et al., 2010; Karlsdöttir et al., 2017; Wen et al., 2018).

Corrosion inhibitors are being investigated as a means of reducing the propensity of corrosion in metallic pipes. Studies on the inhibition of corrosion of metals using synthetic, natural and organic compounds have been reported in the literature. The synthetic corrosion inhibitors are 3-nitro-5-(2-amino-1,3,4-thiadiazolyl) nitrobenzene (NATN) (Al-Baghdadi et al., 2018), and 2-Amino-5-nitro-4,6-diarylcyclohex-1-ene-1,3,3-tricarbonitiles (Chandrabhan et al., 2015). Some organic materials including plant extracts such as Water Hyacinth (Babatunde et al., 2019), Andrographispaniculata (Singh et al., 2010), Spirulina platensis (Kamal and Sethuraman, 2012), Ocimum sanctum (Kumpawat et al., 2010), Jasminum nudiflorum Lindl. Leaves (Li et al. 2010), Carica papaya (Okfor et al., 2007), Phyllanthus amarus (okafor et. al. 2008) and Emblicaofficinalis (Saratha and Vasudha, 2010), have been reported as anticorrosion substances in various corrosive environments.

Despite the vast research carried out on corrosion inhibitors, little or no work has been carried out on the mechanism, kinetics and the thermodynamics of the process. Some researchers reported that inhibitors minimise corrosion in metals through Langmuir adsorption process with values for Standard Change in Gibbs Free Energy and the equilibrium constant (Abiola et al., 2009; Kamal et al., 2012; Babatunde et al., 2019). However, the corresponding values of Standard Entropy Change and
Standard Change in Heat of Reaction change were not reported. Thus, the objective of this paper is to investigate experimentally the corrosion inhibitory effect, kinetics and thermodynamics of the adsorption behaviour of *Afraegle Paniculata* extract in acidic medium.

2. Methods and Materials

2.1 Materials

2.1.1 Preparation of Galvanised Steel Metal Specimen

Galvanised steel rod of diameter 0.8cm and thickness 0.02 cm was obtained from local supplier and cut into 1 cm in length. The specimens were cleaned using absolute ethanol, rinsed with double distilled water, dried with acetone and kept in desiccator until they were ready for use. Analytical grade chemicals were used and appropriate concentrations of acids were prepared using distilled water.

2.1.2 Plant extraction and Solution preparation

*Afraegle Paniculata* leaf was dried, ground to powder form, and soaked with ethyl acetate for 72 hours. The extract was filtered, and the filtered solution was concentrated by distillation and the concentrate air-dried. The dried extract was used for the preparation of inhibitor test solutions with specific concentrations (0.025, 0.05, 0.1, 0.2 and 0.4g/L) by dissolving extract in 1M H$_2$SO$_4$

2.1.3 Extract Analysis

Chemical composition of the plant extract was analysed using gas chromatography–mass spectrometry (GS-MS). The analysis was carried out using an Agilent Technology Gas Chromatography System 7890, equipped with non-polar fused silica capillary DB-1 column (30m length, 0.32 mm internal diameter, and thickness 0.25 μm). The oven temperature was set at 80°C to 240°C, programmed to increase at the rate of 10°C/min and finally held isothermally for 10 min. The injector and detector temperatures were kept at 200°C and 250°C, respectively. The carrier gas used was helium at a flowrate of 1 mL/min, and the splitting ratio was set at 100:1. The Agilent Technology gas chromatograph was connected to an Agilent Mass Spectrometer 5975. The Mass spectral ionization temperature was set at 230°C. The mass spectrometer was operated using the electron impact ionization mode at a voltage of 70 eV, and the mass spectra were taken over the m/z range of 30-700 amu. The components of the plant extract were identified by WILEY and NIST database matching and by comparison of mass spectra with published data (Adams, 2001).

2.2 Gravimetric Method

Gravimetric Method, also known as weight-loss measurement, as reported by Abiola et al. (2008, 2007), and (2006), and Babatunde et al. (2019), was used to determine the corrosion reaction rate of galvanised steel in sulphuric acid whereby cleaned steel metal specimens were immersed in different concentrations (1M, 2M, 3M, 4M, and 5M) of sulphuric acid for a period of five days. The specimens were retrieved after every 24 hours from the acidic solutions, cleaned, weighed and returned into the acidic solution. Each retrieved metal specimen was cleaned following the ASTM G1 Standard (1999), whereby each retrieved metal specimen was immersed in cleaning reagent (50g of NaOH, 200g of Zn dust in 1000 mL H$_2$O) at 90°C for 50 minutes. Thereafter, the corrosion product was further removed by scrubbing of the metal under running water, after which it was dried with acetone and then reweighed. Gravimetric method was also used to determine the corrosion reaction rate of galvanised steel in sulphuric acid with inhibitors, whereby cleaned steel metal specimens were immersed in different solution of 1M H$_2$SO$_4$ containing different concentrations of *Afraegle Paniculata* extract (0.025, 0.05, 0.1, 0.2 and 0.4 g/l). Gravimetric method was used to validate the results of the Gasometric method.

2.3 Gasometric Method

Gasometric analysis as reported by Onuchukwu (1998), Okafor et al. (2008), and Babatunde et al. (2019) was also used to evaluate the corrosion rate of steel via gas evolution. Clean specimen of galvanised metal pipe was fully submerged into 100 ml of test solutions containing 1M H$_2$SO$_4$ with the five different concentrations of *Afraegle Paniculata* extract (0.025, 0.05, 0.1, 0.2 and 0.4 g/l). The test solution was transferred into the reaction vessel, which was connected to a manometer. The initial manometer reading prior to the experiment was taken, after which subsequent manometer readings were taken every 10 seconds for a period of 3 minutes during each experimental run that is after the cleaned metal specimen was dropped into the solution and the reaction vessel was quickly closed. Volume of hydrogen gas evolved was measured and the experiment was carried out at 30, 40, 50, 60 and 70°C. The blank experiment was performed under the same conditions. Each experiment was repeated twice for validation of the results.

2.4 Surface Analysis - Scanning Electron Microscopy

The surface morphologies of the exposed specimens (un-corroded, corroded and corroded with inhibitors metal samples) were carried out using a Tescan VEGA3 scanning electron microscope (SEM) and a Bruker energy dispersive X-ray spectroscopy (EDS) system. Imaging was performed at an accelerating voltage of 15 kV using a secondary electron detector (SEI), and magnifications of 100, 250, 500, 750 and 1000x. For the un-corroded sample, a cleaned steel specimen of 1 cm in length, 0.8cm in diameter and 0.02cm in thickness was used for the analysis. For the corroded specimens without inhibitor, a steel specimen of 1 cm in length, 0.8 cm in diameter and 0.02 cm in thickness
was immersed for 72 hrs in 100 mL of 0.5M H2SO4 solution; while for the characterisation of the corroded specimens with inhibitor, a steel specimen of similar geometry was immersed for 72 hrs in 100mL of 0.5M H2SO4 solution containing Afraegle Paniculata extract.

3. Results and Discussion

3.1 Characterisation of Afraegle Paniculata Extract

The corrosion inhibitive action of phytochemicals has been attributed to the presence of heteroatoms, conjugated or non-conjugated pi bond on the phytochemicals. Lone pair of electrons on heteroatom and delocalized electron on pi bond are regarded as active site of adsorption process by forming coordinate bond with vacant d-orbitals of metal atoms. (De Souza and Spinelli, 2009; Abdel-Gaber et al., 2009).

GC-MS analysis of A. Paniculata extract revealed 27 components, which are all oxygenated compounds (see Table 1). All the major components of the extract contained pi electron in addition to heteroatoms on their molecules. Thus, the corrosion inhibitive action of Afraegle Paniculata extract observed in this study could be attributed to the adsorption of heteroatom or pi electron containing components onto the metal surface.

Table 1. Composition of Afraegle Paniculata Extract generated from the Chromatogram

<table>
<thead>
<tr>
<th>S/N</th>
<th>NAME</th>
<th>Molecular Formula</th>
<th>Rt</th>
<th>% Composition</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Caryophyllene oxide</td>
<td>C15H24O</td>
<td>11.344</td>
<td>0.42</td>
</tr>
<tr>
<td>2</td>
<td>Eudesma-4(15),7-dien-1-[β]-ol</td>
<td>C15H24O</td>
<td>11.777</td>
<td>0.93</td>
</tr>
<tr>
<td>3</td>
<td>10,10-Dimethyl-2,6 dimethylenecyclo[7,2,0]undecan-5-[β]-ol</td>
<td>C15H24O</td>
<td>11.863</td>
<td>0.66</td>
</tr>
<tr>
<td>4</td>
<td>2-Pentadecanone, 6,10,14-trimethyl</td>
<td>C16H32O</td>
<td>13.868</td>
<td>0.44</td>
</tr>
<tr>
<td>5</td>
<td>Dihydrofuran(3,2-F)Coumaranone</td>
<td>C12H8O4</td>
<td>14.435</td>
<td>0.90</td>
</tr>
<tr>
<td>6</td>
<td>n-Hexadecanoic acid</td>
<td>C16H32O2</td>
<td>14.392</td>
<td>3.09</td>
</tr>
<tr>
<td>7</td>
<td>Methoxsalen</td>
<td>C12H8O4</td>
<td>15.063</td>
<td>0.86</td>
</tr>
<tr>
<td>8</td>
<td>7H-Furo[3,2-g][1]benzopyran-7-one, 4methoxy-</td>
<td>C12H8O4</td>
<td>15.330</td>
<td>0.67</td>
</tr>
<tr>
<td>9</td>
<td>Heptadecanoic acid</td>
<td>C17H34O2</td>
<td>15.435</td>
<td>3.21</td>
</tr>
<tr>
<td>10</td>
<td>Phytole</td>
<td>C18H36O</td>
<td>16.154</td>
<td>0.68</td>
</tr>
<tr>
<td>11</td>
<td>9-Octadecenoic acid</td>
<td>C18H34O2</td>
<td>16.287</td>
<td>0.58</td>
</tr>
<tr>
<td>12</td>
<td>7H-Furo[3,2-g][1]benzopyran-7-one, 4,9-dimethoxy-</td>
<td>C18H34O2</td>
<td>16.711</td>
<td>0.58</td>
</tr>
<tr>
<td>14</td>
<td>Sesquirosafuran</td>
<td>C18H30O</td>
<td>18.044</td>
<td>2.39</td>
</tr>
<tr>
<td>15</td>
<td>alpha.-Amyrin</td>
<td>C30H50O</td>
<td>18.139</td>
<td>3.47</td>
</tr>
<tr>
<td>16</td>
<td>4,4,6a,6b,8a,11,11,14b-Octamethyl-2,4a,5,6,7,8,9,10,12a,14a-dodecachydro-1H-picen-3-one</td>
<td>C15H23O</td>
<td>18.292</td>
<td>26.55</td>
</tr>
<tr>
<td>17</td>
<td>p -(3-Methyl-5-oxo-2-pyrazolin-1-yl)benzoic acid</td>
<td>C11H10N2O3</td>
<td>18.506</td>
<td>3.93</td>
</tr>
<tr>
<td>18</td>
<td>2H-1-Benzopyran-2-one, 7-[[(3,7-dimethyl-2,6-octadienyl)oxy]-, (E)-</td>
<td>C19H22O3</td>
<td>19.249</td>
<td>0.37</td>
</tr>
<tr>
<td>19</td>
<td>7-Isopropenyl-1,4a-dimethyl-4,4a,5,6,7,8-hexahydro-3H-naphthalen-2-one</td>
<td>C16H30O</td>
<td>19.644</td>
<td>2.29</td>
</tr>
<tr>
<td>20</td>
<td>2(1H)Naphthalene, 3,5,6,7,8,8a-hexahydro-4,8a-dimethyl-6-(1-methylthienyl)-</td>
<td>C18H30O</td>
<td>18.687</td>
<td>7.91</td>
</tr>
<tr>
<td>21</td>
<td>Lupeol</td>
<td>C20H30O</td>
<td>18.763</td>
<td>9.15</td>
</tr>
<tr>
<td>22</td>
<td>7,22-Ergostadienol</td>
<td>C28H46O</td>
<td>18.839</td>
<td>0.94</td>
</tr>
<tr>
<td>23</td>
<td>2H-1-Benzopyran-2-one, 7-[3,7-dimethyl-2,6-octadienyl]oxy]-, (E)-</td>
<td>C19H20O</td>
<td>19.249</td>
<td>0.37</td>
</tr>
<tr>
<td>24</td>
<td>2,6,11-Dodecatrienal, 2,6,10-trimethyl-, (E,E,E)-</td>
<td>C18H30O</td>
<td>19.368</td>
<td>0.47</td>
</tr>
<tr>
<td>26</td>
<td>9,19-Cycloergost-24(29)-en-3-ol, 4,14-dimethyl-, acetate, (3β,4α,5α)-</td>
<td>C23H36O4</td>
<td>19.968</td>
<td>1.55</td>
</tr>
<tr>
<td>27</td>
<td>Curan-17-oic acid, 2,16-didehydro-20-hydroxy-19-oxo-, methyl ester</td>
<td>C20H22N2O4</td>
<td>20.177</td>
<td>1.27</td>
</tr>
<tr>
<td>28</td>
<td>Hop-22(29)-en-3.beta.-ol</td>
<td>C18H30O</td>
<td>20.353</td>
<td>2.49</td>
</tr>
</tbody>
</table>

3.2 Gravimetric Analysis

3.2.1 Corrosion Reaction Rate Kinetics.

The weight loss was calculated in grams as the difference between the initial weight prior to immersion, and weight after removal of the corrosion product.

\[ \Delta W = W_i - W_f \]  \hspace{1cm} (1)

3.2.2 Steel at different concentration of H2SO4 after 24 Hours

As showed in Figure 1, the weight loss of the steel metal increased with an increase in the concentration of the H2SO4 and the largest weight loss occurred after the first 24 hours. This can be ascribed to the decrease in the corrosion reaction rate after the first 24 hours (i.e. the initial reaction rate). In Figure 2, it shows that the initial reaction weight loss (first 24 hours) was directly proportional to the concentration. The initial mass loss was used to calculate the time and space average corrosion reaction rate, which was determined using the same methodology as reported by Belarbi et al. (2018):
\[-r_{\text{CR}} = \frac{(K \times \Delta W)}{(A \times t \times \rho)} \]  
(2)

Where \(-r_{\text{CR}}\) is the corrosion rate in mm/y, \(K\) is the conversion factor \(8.76 \times 10^4 = 24 \, \text{h/d} \times 365 \, \text{d/y} \times 10\, \text{mm/cm}\), \(\Delta W\) is the weight loss in g, \(A\): area in cm², \(t\) is the time of exposure in h, \(\rho\) is the density of steel, 7.87 g/cm³.

\[\ln (-r_{\text{CR}}) = \ln (k) + \alpha \ln (C) \]  
(3)

Where \(k\) is the Corrosion reaction constant in \(\text{mm} \, \text{dm}^3/\text{yr} \, \text{mol}\), \(\alpha\) is the order of reaction, and \(C\) is the Concentration of acidic solution in mol/dm³.

Figure 3 showed that the average corrosion reaction rate was directly proportional to the sulphuric acid concentration, while Figure 5 confirmed that the corrosion reaction followed first order kinetics. These findings were also reported by Babatunde et al. (2019). Furthermore, from Figure 4, the corrosion reaction rate kinetic constant \((k)\) was deduced to be 23.7 mm day⁻¹ M⁻¹.

### 3.2.3 Inhibition Efficiency of Afraegle Paniculata

The method reported by Abiola et al. (2009) and Babatunde et al. (2019) was used to determine the percentage inhibition efficiency (% I) as follows:

\[I\% = \left(\frac{W_u - W_i}{W_u}\right) \times 100 \]  
(4)

Where \(I\%\) is the inhibition efficiency of Water Hyacinth in %, \(W_u\) is the weight loss of uninhibited steel metal in acidic solution, and \(W_i\) is the weight loss of inhibited steel metal in acidic solution with Afraegle Paniculata extract.

Figure 5 showed that truly Afraegle Paniculata inhibited corrosion of steel by sulphuric acid and the percentage inhibition efficiency increased with increasing extract concentration, but the rate of increase dropped with time after the first 24 hours (i.e. initial reaction).

It was confirmed that the increase in the extract concentration raised the number of inhibitor molecules that were adsorbed onto the steel surface, thus, preventing the adsorption of corrosion species (ions) in the acidic solution. In other words, the adsorption of the inhibitor molecules decreased the number of available active sites, that is, surface area for the corrosion species; thus reducing the rate of corrosion.
The inhibition efficiency at the initial reaction stage (i.e. 24 hours as shown in Figure 6) was used to determine the degree of surface coverage ($\theta$) and the adsorption isotherm. The degree of surface coverage ($\theta$) was deduced as reported by Abiola et al. (2009), Singh et al. (2011) and Babatunde et al. (2019).

$$\theta = \frac{I\%}{100}$$  \hspace{1cm} (5)

### 3.2.4 Adsorption Isotherm

Data of degree of surface coverage were fitted into different isotherms and it was deduced that adsorption relationship follows the Langmuir Adsorption model (see Figure 8), which can be expressed as

$$\frac{C_{\text{inh}}}{\theta} = \frac{1}{K} + C_{\text{inh}}$$ \hspace{1cm} (6)

where $C_{\text{inh}}$ is the inhibitor concentration and $K$ the equilibrium constant for the adsorption/desorption process of the inhibitor molecules on the metal surface.

Using the equation that describes Langmuir adsorption isotherm i.e. Equation 6, the equilibrium constant for the adsorption/desorption process ($K$) was deduced as the inverse of the intercept of Figure 7. The adsorption equilibrium constant was used to deduce the standard change in Gibbs Free Energy of adsorption expression as reported by Abiola et al. (2004), Qiu et al. (2005) and Babatunde et al. (2019) (i.e. Equation 7).

$$K = \frac{1}{5.5} \exp \left( - \frac{\Delta G_{\text{ads}}^0}{RT} \right)$$  \hspace{1cm} (7)

Values of the adsorption equilibrium constant and the Standard Change in Gibbs Free Energy of adsorption are shown in Table 2. The value of the Standard Change in Gibbs Free Energy estimated in the present was close to those reported in other studies (Abiola et al., 2004; Qiu et al., 2005; Babatunde et al., 2019). Furthermore, the negative value of the Standard Change in Gibbs Free Energy of adsorption ($\Delta G_{\text{ads}}^0$) showed that the adsorption process was spontaneous and exothermic.

### 3.3 Gasometric Analysis

#### 3.3.1 Corrosion Reaction Mechanism

It was showed in Figures 8 and 9 that the volume of hydrogen gas produced decreases with increasing concentration of inhibitor i.e. *Afraegle Paniculata*. This was due to the decrease in the corrosion reaction rate due to the decrease in the number of active sites available for the corrosion attack. The same observation was reported in the literature by Abiola et al. (2007), Abiola et al. (2009), and Babatunde et al. (2019).
3.3.2 Inhibition Efficiency of Afraegle Paniculata

The volume of hydrogen gas evolved data were used to determine the percentage inhibition efficiency (%I') using Equation 8.

\[ I'\% = \left( \frac{V_a - V_i}{V_a} \right) \times 100 \]  

(8)

Where \( I' \) is the inhibition efficiency of Afraegle Paniculata in %, \( V_a \) and \( V_i \) are Volume of H₂ gas evolved in the absence and presence of extracts, respectively.

Figure 10 showed that the inhibition efficiency increases with increasing Afraegle Paniculata extract concentration. This confirms that an increase in the inhibitor concentration increases the number of its molecules that gets adsorbed onto the steel surface, which in turn reduces the available surface area for the acidic molecule.

3.3.3 Adsorption Isotherm

The inhibition efficiency data were used to determine the degree of surface coverage using the same methodology that was used for the gravimetric analysis. The surface coverage data were further fitted into different isotherms to determine the adsorption isotherm. This was carried out for the various investigated temperatures i.e. 30, 40, 50, 60 and 70°C. It was deduced that the adsorption relationship, irrespective of the temperature of the reaction, follows similar pattern to that of the gravimetric analysis, i.e., the Langmuir Adsorption model, which is described by Equation 6.

3.3.4 Thermodynamic Properties

The thermodynamic properties (Standard Change in Heat of Reaction, Standard Entropy Change and Standard Change in Gibbs Free Energy) were deduced via gasometric analysis whereby each adsorption isotherm was used in calculating the reaction equilibrium constant, which was further used to calculate the Standard Change in Gibbs Free Energy of adsorption \( (\Delta G^\circ_{ads}) \) as previously explained for each temperature considered.

The reaction kinetics parameters shown in Table 3 were used to determine the Standard Change in Heat of Reaction and Standard Entropy Change of the corrosion reaction. The change in Standard Change in Gibbs Free Energy at constant temperature is expressed as Equation 9 below.

\[ \Delta G = \Delta H + T\Delta S \]  

(9)

<table>
<thead>
<tr>
<th>T (°C)</th>
<th>Intercept</th>
<th>K</th>
<th>( \Delta G ) (kJ/mol)</th>
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</table>
It can be deduced from Figure 11 that the Standard Change in Heat of Reaction was -10.88 kJmol⁻¹ while the Standard Entropy Change was -0.139 kJ mol⁻¹K⁻¹. The negative Standard Change in Heat of Reaction value showed that the reaction was an exothermic reaction as energy was lost to the surrounding, resulting in the products having a lesser amount of energy. It also confirmed the spontaneity and thermodynamic feasibility of the reaction. Naturally, when heat is added to a system, its molecules would become more active and the randomness of the molecules will increase which would be shown as an increase in the entropy of the system.

However, when heat is removed from a system, the molecules will be less active and will exhibit less chaotic movement, leading to a decrease in the entropy of the molecules, which would be exhibited as a negative entropy change.

Figure 11. Thermodynamic Properties of Galvanised steel corrosion in 1M H₂SO₄ solution in the presence of different concentrations of Afraegle Paniculata extracts

The negative value of the entropy change also confirms the exothermic nature of the corrosion process. In addition, the standard entropy change of a system depends on the physical state of the system. For example, the entropy of gases is higher than that of solids because an atom in gas phase has more options for position than the same atom in a solid phase. Hence, the negative value of the Standard Entropy Change confirmed the change of phase of the system as hydrogen sulphide gas molecules dissolved in water, forming the acidic solution which reacts with the iron specie to produce iron sulphate. The phase change exhibited during the process was gas to liquid, and finally solid phase.

3.4 Surface Analysis - Scanning Electron Microscopy

The SEM images of the surface of the un-corroded, corroded and inhibited corroded specimens (i.e., at a magnification of 100×, 250×, 500×, 750× and 1000×) were in agreement with the conclusions of the gravimetric and gasometric analysis that steel metal corrodes in acidic environment and Afraegle Paniculata inhibits the corrosion propensity by acting as an inhibitor. The SEM micrographs of the inhibited corroded steel showed the mechanical polishing on the surface of the inhibited corroded steel and this was an indicator that the corrosion on the inhibited steel was minimal. Comparing that to the surface of the un-inhibited corroded steel suggested that the mechanical polished film formed was due to the adsorption of the Afraegle Paniculata molecules.

It was proposed that molecules of the inhibitor and acid adsorbed competitively on the metal surface, and by so doing, affected the morphology and the reaction kinetics of the corrosion products; and the inhibitor molecules adsorption decreases the number of available active sites i.e. surface area for the corrosion species; thus reducing the rate of corrosion attack. Figure 12 confirms the formation of various sizes of pores i.e. cracks due to corrosion, which can lead to the formation of open cracks as the corrosion reaction progresses.

Figure 12. SEM images at a magnification of 250× of the uninhibited corroded steel specimen.

3.4.1 Effect of Cracks

The SEM micrographs in Figure 13 showed that there were larger pores (cracks) sizes on the surface cut-edge section of the inhibited corroded specimen compared to that of the middle section of the same specimen. It can therefore be hypothesized that the presence of cracks on metal surface enhances corrosion, and the corrosion rate is directly proportional to both the number of cracks and the crack sizes. In other words, the higher the number of cracks and the cracks sizes, the higher the rate of corrosion.
3.4.2 Energy Dispersive X-ray Spectroscopy (EDS)
The presence of Sulphur and the absence of Zinc and Aluminium in the EDS analysis of the corroded and inhibited corroded specimens (see Figures 15 and 16), compared to that of the un-corroded specimen (see Figure 14) confirm that galvanised steel corrodes in acidic environment. The weight percentage composition of Iron in the inhibited corroded specimen (see Figure 16) was higher than that of the un-inhibited specimen (see Figure 15); thus, suggesting that Afraegle Paniculata acts as an inhibitor in inhibiting sulphuric acid corrosion of steel.
4. Conclusion
The study investigated the corrosion inhibition properties of *Afraegle Paniculata* extract in inhibiting the corrosion propensity of galvanised steel in acidic solution, with a view to understanding the corrosion reaction mechanisms, adsorption behaviour of *A. Paniculata* and the thermodynamics of the reaction. The experimental investigation was conducted using weight-loss (gravimetric analysis) and gasometric analyses. The surface morphologies of the exposed steel were studied using scanning electron microscope (SEM) and energy dispersive X-ray spectroscopy (EDS) system.

Drawn from the study, it could be concluded that:
1. Galvanised steel was corroded in sulphuric acid environment and the corrosion reaction followed a first order reaction kinetics with a corrosion reaction rate kinetic constant (k) of 23.7 mm day⁻¹ M⁻¹. The corrosion reaction was a spontaneous exothermic reaction.

2. *Afraegle Paniculata* extract was a good inhibitor for galvanised steel in sulphuric acid solution and its inhibition efficiency increased with increasing extract concentration.

3. The adsorption of *Afraegle Paniculata* extract on galvanised steel surface obeyed Langmuir adsorption isotherm with Standard Change in Gibb's Free Energy of adsorption (\(\Delta G_{ads}^o\)) and equilibrium constant at different temperatures determined (see Table 4). The Standard Change in Gibb's Free Energy of adsorption (\(\Delta G_{ads}^o\)) at different temperatures confirmed the thermodynamic feasibility of the reaction.

4. The Standard Change in Heat of Reaction (\(\Delta H_{ads}^o\)) was -10.88 kJ mol⁻¹ and the Standard Entropy Change of reaction (\(\Delta S_{ads}^o\)) was -0.139 kJ mol⁻¹K⁻¹. Both values confirmed the exothermic nature of the reaction.

5. The SEM images confirmed the corrosion of galvanised steel metal in acidic environment, and the corrosion inhibitory properties of *Afraegle Paniculata* via forming a mechanical polished film due to the adsorption of the *Afraegle Paniculata* molecules. The SEM images showed the formation of various sizes of cracks which could lead to open cracks on galvanised steel due to corrosion. The SEM images further confirmed that cracks on metal surface enhance corrosion, and the corrosion rate is directly proportional to both the number of crack and the cracks’ sizes.

6. EDS analysis confirmed that galvanised steel corrodes in acidic environment and the corrosion propensity can be reduced using *Afraegle Paniculata*, which acted as an inhibitor to corrosion attacks of sulphuric acid.

References:


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Effects of Micro Silica and Waste Glass on the Rheological and Mechanical Properties of Self-Compacting Concrete

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Abstract: Self-compacting concrete was produced by partially substituting cement for micro silica (0-15% at an interval of 5%) while waste glass was included in the matrix as part replacement for river sand (10-40% at an interval of 10% by weight of river sand) in each of the micro silica (MS) inclusion (5-15%). The rheological characteristics of the concrete were evaluated using slump flow, T50cm, V-funnel and L-box test while the compressive strength was determined at 7, 14, 21, 28 and 56 days curing by crushing. The total cubes (100 x 100 x 100 mm) that were cast, cured and crushed stood at 240. The slump flow decreased with increased inclusion of MS, T50cm and V-funnel flow time increased with increased replacement, while L-box values decreased indicating reduction in passing ability, this is due to the high water absorption capacity of MS. The reverse was the case when waste glass (WG) was introduced into the concrete matrix due to the low water absorption capacity of WG compared to river sand as evident in water absorption test. At 15% MS the passing ability values (0.76 for SCC0 and 0.79 for 10% WG) were below the EFNARC (2002) standard based on the L-box test conducted. The compressive strength at 56 days in percentage of the control were; 19.58%, 28.90%, and 23.77% for 5%-15% MS replacement without WG, respectively. Thus, MS can be blended with cement up to 15% with appreciable gains in compressive strength with optimum replacement at 10%. As the WG was introduced, there was loss in compressive strength. The variation in compressive strength in percentage of the control for 10-40% replacement of river sand with WG were; +21.21%, +13.52%, 0.23% and -18.41% for 5% MS, +23.54%, +16.55%, +11.89%, and +4.9% for 10% MS, +16.55%, +11.19%, 0.70% and -0.11% for 15% MS, all at 56 days. Based on EFNARC (2002) and the compressive strength, 10% MS has the best rheological characteristics.

Keywords: Self-compacting concrete, micro silica, waste glass, rheological characteristics, compressive strength

1. Introduction

Globally, more than 10 x 10^9 tons of concrete are made annually (Mehta, 1999; Malhotra, 2000). In order to produce over 10 billion tons of concrete annually, large amount of cement would be required. The volume of cement produced globally had risen to 3 billion tons in 2012 from 1,500 Million tons in 2000 (Imbabi et al., 2012). In normal concrete production, about 12% of cement, 8% water (for mixing) and 80% aggregates (fine and coarse aggregate), all measured by mass are required (Mehta, 2001). This implies that, the concrete industry consumes over 9 billion tons of aggregate (fine and coarse), and 1 billion tons of water (for mixing) annually at a global 1.5 billion tons of cement utilisation in concrete production per year. This huge production of cement contributes about 5-7% of total CO2 emission (Turner and Collins, 2013). This emission activates greenhouse effect. Thus, it is expedient to reduce this emission. One of the useful means of achieving this is by utilising industrial by-products and waste materials, as partial substitute for cement (Kartini et al., 2014; Crossin, 2015; Yang et al., 2015).

Industrial by-products and waste materials (such as, fly ash, granulated blast furnace slag, rice husk ash, micro silica, and etcetera) are not themselves binders but are pozzolanic materials that will in finely divided form, react chemically with lime produced during the hydration of cement to form cementitious material. This reaction which takes place at ordinary temperature leads to reduction in the heat of hydration and thus, reduction in cracking is achieved. In this research, densified micro silica (MS) was utilised.

Excessive mining of natural resources for aggregates lead to some environmental implications which include; destructions of wetlands and mangrove marshes, bank erosion, turbidity of water-body, disappearance of some phytoplankton and zooplankton including many kinds of macro-benthos which ultimately affects the distribution of fishes (Adekunbi et al., 2018). Not all glasses are recyclable, especially when colours are mixed up. This mixing affects the chemical composition of waste glass during recycling. Since glasses are non-biodegradable, using it for landfill would constitute environmental challenges.
In recent times, interest in self-compacting concrete (also called self-consolidating concrete) has grown. This type of concrete requires high quantity of cement than vibrated concrete for the same compressive strength. However, the cement content can be reduced by the introduction of MS as partial substitute for cement. The addition of MS to a concrete mix leads to increase in strength by 30-100%, depending on the kind of mix, category of cement, use of plasticizers, nature aggregate and curing methods. Some researchers (Sellevold and Radjy, 1983; Faseyemi, 2012; Vivek and Dhinakaran, 2015) observed that at 10% replacement of cement with silica fume compressive strength was more than the control. Other studies (e.g., Navaneethakrishnan and Shanthi, 2012; Barisua et al., 2019) found that the utmost compressive strength was at 15% substitution of cement in lieu of micro silica. As cement substitution increases, the slump flow reduced, the time of flow for V-funnel, V-funnel T5minutes and slump flow increased indicating increase in viscosity (Gowda et al., 2016). Figure 1 shows a typical setup of V-funnel and slump flow tests.

Vikraman and Nithya (2015) observed that the introduction of waste glass (WG) as partial substitute for fines in a self-compacting concrete containing fly ash (10% replacement) affected rheological and hardened properties of the concrete. At 40%-50% replacement of fines with WG, the slump flow values were above the recommended requirements of EFNARC (2002), with slump flow value reaching 860 mm and 890 mm, respectively. There was loss in compressive strength at all ages with increase in WG content. Moreover, Sameer et al. (2016) reported a decrease in compressive strength for grade 20, 25 and 30 concrete, when fine aggregate was partially substituted with waste glass at 5%, 10% and 15%, while fly ash content was kept constant at 20%. The percentage downturn in compressive strength as fine aggregates was replaced in percentages of 5%-15%, with respect to the control (CM) at 28 days were; -8.2%, -15.4%, -25.1% for grade 20 concrete; -3.4%, -11.3% and -17.8% for grade 25 concrete; -6.6%, -13.9% and -17.7% for grade 30 concrete. Similar effect was observed by Buari et al. (2019), using WG as partial substitute for fines in a SCC blended with groundnut shell ash. Abin et al. (2017) conducted a study on the replacement of river sand with iron ore as fines in self-compacting concrete. The substitution ranged from 0% to 100% at regular intervals of 10%. The slump flow diped with increase in percentage replacement of river sand with iron ore fines. The compressive strength improved with increase in replacement unto 40%, after which, the strength started reducing.

In all the replacements, the compressive strengths were beyond the control mix except for 100% which was below the control. For Kumar et al. (2016), the Compressive strength improved with increase in percentage replacement of fine aggregate with quarry dust up to 30% substitution. Percentage substitution of cement for fly ash was kept constant at 30%.

Little or no work has been done in the development of green concrete using MS and WG. This research is geared towards the development of green and cost-effective concrete using waste materials with acceptable rheological properties and better compressive strength than the control.

2. Materials and Methods

The investigations were laboratory experiment based. The following constituents were utilised in this research work; Cement, Micro silica, Coarse aggregate (50% 12.5mm and 50% 10 mm granite), Fine aggregate (River sand), Waste glass, Water, and Superplasticizer.

The cement (Dangote’s brand of grade 42.5 CEM II Portland cement) used for the preparation of concrete specimens in this research was obtained from mile (III) building material market in Port Harcourt Rivers state Nigeria, conformed with BS 197-1:2000 (BSI, 2000).

Micro Silica (MS) was subjected to chemical test to determine its chemical composition and also its reactivity. Chappelle’s reactivity test was conducted on the MS in accordance with NF P18-513:2010, at Segun Olabiran and Associates laboratory old Ife road Ibadan Nigeria. Cement was substituted for MS in percentages of 0%, 5%, 10% and 15%. The MS was bought from Alibaba online shop.

The waste glass was obtained from a glass dealer shop at No.26 Azikiwe Street Mile (III) Diobu Port Harcourt, Rivers State, Nigeria. The material was washed, dried and ground to obtain glass cullet. The cullet was sieved. Only those passing through sieve 4.75mm and retained on sieve 0.150 mm was used. The waste glass was added in percentages of 10%, 20%, 30%, and 40% of the sand replacement. The chemical composition of the WG and MS was evaluated at DE-image Laboratory Services, Ibadan.
River sand free from impurities was used for the research. Only river sand passing through sieve 4.75mm but retained on 0.150 mm was used. The river sand was obtained from Choba river sand dredging site in Port Harcourt Rivers state Nigeria. Crushed rough textured granite obtained from Crushed Rock Industry Nigeria limited, Akamkpa in Cross Rivers State, Nigeria, served as coarse aggregate in the research. Its size was made up of 12.5 mm (50%) and 10 mm (50%) only. The material was washed to remove impurities that may affect its intended use in accordance with BS 882:1992 (BSI, 1992).

Moreover, water used for mixing and curing was obtained from the laboratory and met the requirements of BS EN 1008:2002 (BSI, 2002a). The distilled water used for chappelles test was obtained from the department of zoology university of Ibadan, Oyo State, Nigeria. Fosroc Conplast SP430 conforming to BS 5075-1:1982 (BSI, 1982), was used as superplasticizer to improve the rheological properties of the concrete. A summary of the various test conducted and the standards adopted are presented in Table 1.

### 3. Mix design
The EFNARC (2002) was adopted as a guide for the mix design. A synopsis of the various constituent is displayed in Table 2.

### 4. Results and Discussion

#### 4.1 Reactivity and Chemical Composition Test
According to NF P18-513:2010 (NF, 2020), the quantity of Ca(OH)₂ depletion by the pozzolanic material should exceed 600 mg/g, for such sample to be regarded as reactive. Since the value so obtained (1267.1 mg/g) as shown in Table 3 is greater than 600 mg/g, the material is pozzolanic. From the Chemical composition test results shown in Table 4, the summation of Fe₂O₃, SiO₂ and Al₂O₃ for MS is 94% which is 35.7% greater than the 70% ASTM C618 recommended standard (ASTM, 1994a). Therefore, the material met the requirement for use as pozzolanic material in accordance with ASTM C618 (ASTM, 1994a).

<table>
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<tr>
<th>Experiments</th>
<th>Sample</th>
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<th>5%MS Mix A</th>
<th>10%MS Mix B</th>
<th>15%MS Mix C</th>
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Table 2. Mix Proportion for Various Replacement for 1 m³ of Concrete

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<th>Mix</th>
<th>Cement kg/m³</th>
<th>Micro Silica kg/m³</th>
<th>Fine Aggregates Kg/m³</th>
<th>Glass cullet Kg/m³</th>
<th>Coarse aggregates kg/m³</th>
<th>Water kg/m³</th>
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<td>77.1</td>
<td>492.6</td>
<td>268.4</td>
<td>180</td>
<td>10.28</td>
<td></td>
</tr>
</tbody>
</table>

**Keys:** SCC= Self-Compacting Concrete
MS=Micro silica
WG=Waste Glass
CM = No WG and MS
SCC0% = MS without WG
SCC10% = MS and 10%WG
SCC20% = MS and 20%WG
SCC30% = MS and 30%WG
SCC40% = MS and 40% WG

Table 3. Reactivity and Physical Properties of Materials

<table>
<thead>
<tr>
<th>Parameters</th>
<th>Silica fumes</th>
<th>Waste glass</th>
<th>River sand</th>
<th>Coarse aggregate</th>
</tr>
</thead>
<tbody>
<tr>
<td>Chappelle’s reactivity test Ca(OH)₂ fixed mg</td>
<td>1267.1</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Fineness modulus (FM)</td>
<td>-</td>
<td>2.98</td>
<td>3.72</td>
<td>-</td>
</tr>
<tr>
<td>Coefficient of uniformity Cₜ</td>
<td>-</td>
<td>2.98</td>
<td>3.72</td>
<td>-</td>
</tr>
<tr>
<td>Coefficient of curvature Cc</td>
<td>-</td>
<td>1.25</td>
<td>1.18</td>
<td>-</td>
</tr>
<tr>
<td>Bulk density Kg/m³</td>
<td>-</td>
<td>1485</td>
<td>1554</td>
<td>1658</td>
</tr>
<tr>
<td>Specific gravity</td>
<td>2.20</td>
<td>2.34</td>
<td>2.49</td>
<td>2.70</td>
</tr>
<tr>
<td>Water absorption (%)</td>
<td>-</td>
<td>0.49</td>
<td>1.07</td>
<td>1.23</td>
</tr>
</tbody>
</table>

Table 4. Chemical Constituents of MS and WG

<table>
<thead>
<tr>
<th>ID</th>
<th>% SiO₂</th>
<th>% Al₂O₃</th>
<th>% Fe₂O₃</th>
<th>% CaO</th>
<th>% Na₂O</th>
<th>% MgO</th>
<th>% K₂O</th>
<th>% LOI</th>
</tr>
</thead>
<tbody>
<tr>
<td>MS</td>
<td>91.214</td>
<td>0.635</td>
<td>0.315</td>
<td>0.460</td>
<td>0.721</td>
<td>0.413</td>
<td>0.123</td>
<td>3</td>
</tr>
<tr>
<td>GLASS</td>
<td>72.216</td>
<td>9.010</td>
<td>0.0210</td>
<td>0.0025</td>
<td>0.030</td>
<td>0.009</td>
<td>0.0034</td>
<td>-</td>
</tr>
</tbody>
</table>

4.2 Physical Properties of Materials

The ASTM C127-15 (ASTM, 1994b) stipulates that the bulk density of aggregates for the production of normal weight concrete should be 1120-1920 Kg/m³. Thus, the aggregates met the requirement as shown in Table 3. The specific gravity of MS was within the range specified by ACI Committee 232 (ACI, 2000) which specified a range of 2.1-2.3 micro silica. The specific gravity of the river sand, WG and coarse aggregates were; 2.34, 2.49 and 2.7, respectively. Figure 2 depicts a sieve analysis curve for fine aggregates (river sand and waste glass). These values were within the recommended values of 2.20-3.00 as stipulated by ASTM C33 / C33M – 18 (ASTM, 1994c) for normal weight aggregates. The water absorptions values fell within the recommended limit given in BS 6349-2:2019 (BSI, 2019) which specified a maximum water absorption of 2-3%.

4.3 Rheological Characteristics

The Rheological Characteristics were evaluated using slump flow, T₅₀cm, v-funnel and L-box test. It was observed that as MS was introduced, the slump flow reduced, v-funnel flow time increased, as well as T₅₀cm time, while the
L-box value decreased (decrease in L-box value indicate low passing ability) as shown on Table 5 and Figures 3-6. This is because the micro silica absorbed more water leading to increased viscosity. This is in tandem with the works of other researchers, viz: Gowda et al. (2016) and Obunwo et al. (2018).

However, as the WG were introduced in percentages, the reverse was the case. This is due to the fact that the WG absorbed lesser quantity of water compared to the river sand used as evident in the water absorption test conducted. This phenomenon is similar to the findings of Buari et al. (2019). However, this result is in contrast with the findings of Abin et al. (2017) who observed that slump flow decreased with increased replacement of fine aggregates. In their research, iron filling was used as partial replacement for fines blended with fly-ash. Thus, the variance in the rheological properties is connected with water absorption capacity of replacing materials.

The slump flow, T50 cm and V-funnel flow were within the EFNARC (2002) standard for rheological properties. However, the L-box values for MIX C 15% MS (Mix ID SCC0 and SCC10% WG) were below the EFNARC (2002) as shown in Table 5. The various mix that met the EFNARC (2002) standard, can be used for different concrete works depending on the need; normal applications (e.g. walls, columns), very congested reinforced structures, structures with complex shapes, or for filling under formwork.

<table>
<thead>
<tr>
<th>Mix ID</th>
<th>Slump Flow (mm)</th>
<th>Slump Flow T50 (sec)</th>
<th>V-Funnel Flow (sec)</th>
<th>L-Box h2/h1</th>
</tr>
</thead>
<tbody>
<tr>
<td>CM</td>
<td>690</td>
<td>2.98</td>
<td>7.98</td>
<td>0.83</td>
</tr>
<tr>
<td>SCC0</td>
<td>681</td>
<td>3.40</td>
<td>8.21</td>
<td>0.82</td>
</tr>
<tr>
<td>SCC10% WG</td>
<td>702</td>
<td>3.17</td>
<td>7.92</td>
<td>0.84</td>
</tr>
<tr>
<td>SCC20% WG</td>
<td>716</td>
<td>2.80</td>
<td>7.62</td>
<td>0.86</td>
</tr>
<tr>
<td>SCC30% WG</td>
<td>729</td>
<td>2.46</td>
<td>7.22</td>
<td>0.89</td>
</tr>
<tr>
<td>SCC40% WG</td>
<td>746</td>
<td>2.20</td>
<td>6.78</td>
<td>0.90</td>
</tr>
<tr>
<td>MIX B 10% MS</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>SCC0</td>
<td>665</td>
<td>3.86</td>
<td>9.76</td>
<td>0.80</td>
</tr>
<tr>
<td>SCC10% WG</td>
<td>677</td>
<td>3.50</td>
<td>9.26</td>
<td>0.80</td>
</tr>
<tr>
<td>SCC20% WG</td>
<td>691</td>
<td>3.28</td>
<td>8.56</td>
<td>0.82</td>
</tr>
<tr>
<td>SCC30% WG</td>
<td>708</td>
<td>3.00</td>
<td>7.88</td>
<td>0.85</td>
</tr>
<tr>
<td>SCC40% WG</td>
<td>721</td>
<td>2.75</td>
<td>7.32</td>
<td>0.85</td>
</tr>
<tr>
<td>MIX C 15% MS</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>SCC0</td>
<td>659</td>
<td>3.98</td>
<td>10.99</td>
<td>0.76</td>
</tr>
<tr>
<td>SCC10% WG</td>
<td>664</td>
<td>3.86</td>
<td>10.24</td>
<td>0.79</td>
</tr>
<tr>
<td>SCC20% WG</td>
<td>675</td>
<td>3.60</td>
<td>9.56</td>
<td>0.80</td>
</tr>
<tr>
<td>SCC30% WG</td>
<td>688</td>
<td>3.30</td>
<td>8.80</td>
<td>0.80</td>
</tr>
<tr>
<td>SCC40% WG</td>
<td>703</td>
<td>3.18</td>
<td>8.20</td>
<td>0.81</td>
</tr>
<tr>
<td>Recommended Limits (EFNARC,2002)</td>
<td>650-800</td>
<td>2-5</td>
<td>6-12</td>
<td>0.8-1</td>
</tr>
</tbody>
</table>

Table 5. Rheological Characteristics of Concrete
4.4 Compressive Strength

The outcome of the compressive strength experiments is presented in Table 6 and Figures 7-9. The result indicates that at 10% substitution of cement for MS, the compressive stress was maximum at all ages. This result is in tandem with the finding of Vivek and Dhinakaran (2015). This is not in agreement with the findings of Navaneethakrishnan (2012) and Barisu et al (2019), who observed utmost compressive stress at 15% substitution of cement for micro silica. This difference might be due to any of the following reasons: kind of mix, category of cement, use of plasticizers, nature aggregate and curing methods. The compressive stress at 7, 14, 21, 28 and 56 days were 33, 36.4, 40, 42, and 42.9 MPa for CM; 34, 38.6, 43, 45.6, and 51.3 MPa for 5% MS; 36.8, 42, 47.7, 50 and 55.3 MPa for 10% MS; 33.3, 39.5, 45, 47 and 53.1 MPa for 15% MS, respectively. At 28 and 56 days, the percentage boost in compressive stress were 8.6% and 19.58% for 5% MS; 19.0% and 28.90% for 10% MS, 11.9% and 23.77% for 15% MS, respectively. The MS had positive impact on the compressive strength of the concrete as it aged from 7 to 56 days with a maximum compressive strength of 55.3 MPa at 10% replacement at 56 days. This improvement in compressive strength is due to the pozzolanic activities of MS. As the percentage of WG in the matrix increased from 10%-40%, generally, the compressive strength downturned.

For Mix A, there was rise in compressive strength for 10% substitution of river sand for WG at all ages. Disparities in compressive strength are expressed in percentage of the control (CM) at 7, 14, 21, 28 and 56 days as follows; +7.88%, +9.89%, +10.00%, +11.9% and +21.21% for 10% WG; -3.03%, -1.37%, 0%, +4.76% and +13.52% for 20% WG; -11.52%, -10.44%, -10.25%, -9.52%, and -2.1% for 30% WG; -24.5%, -23.08%, -21%, -21.30% and -18.41% for 40% WG, respectively. When compared with 5% MS without WG, the difference in percentage at 7, 14, 21, 28 and 56 days were: +4.7%, +3.63%, +3.53%, +3.07%, and +1.36% for 10% WG; -5.88%, -6.99%, -5.21%, -3.51% and -5.07% for 20% WG; -14.12%, -15.54%, -15.53%, -16.67% and -18.13% for 30% WG; -26.76%, -27.46%, -25.65%, -27.63% and -31.77% for 40% WG, respectively. Thus, comparing the results with CM and 5% MS0% WG, waste glass can be introduced up to 20% and 10%, respectively with improvement in compressive strength at 56 days.

For Mix B, there was difference in compressive strength as WG was introduced at various percentages. This variation is expressed in percentage of CM at 7, 14, 21, 28 and 56 days as follows; +2.73%, +9.89%, +10.00%, +11.9% and +23.54% for 10% WG; -3.03%, +3.85%, +5.50%, +9.05%, and +16.55% for 20% WG; -3.03%, +3.85%, +5.50%, +9.05%, and +16.55% for 20% WG; -9.09%, +0.55%, +0.75%, +2.38% and +11.89%
Table 6. Compressive Strength of Cubes for All Ages

<table>
<thead>
<tr>
<th>Mix ID</th>
<th>Age (Days)</th>
<th>7</th>
<th>14</th>
<th>21</th>
<th>28</th>
<th>56</th>
</tr>
</thead>
<tbody>
<tr>
<td>CM</td>
<td>33.0</td>
<td>36.4</td>
<td>40.0</td>
<td>42.0</td>
<td>42.9</td>
<td></td>
</tr>
<tr>
<td>SCC0% WG</td>
<td>34.0</td>
<td>36.6</td>
<td>42.5</td>
<td>45.6</td>
<td>51.3</td>
<td></td>
</tr>
<tr>
<td>SCC10% WG</td>
<td>35.6</td>
<td>40.0</td>
<td>44.0</td>
<td>47.0</td>
<td>52.0</td>
<td></td>
</tr>
<tr>
<td>SCC20% WG</td>
<td>32.0</td>
<td>35.9</td>
<td>40.0</td>
<td>44.0</td>
<td>48.7</td>
<td></td>
</tr>
<tr>
<td>SCC30% WG</td>
<td>29.2</td>
<td>32.6</td>
<td>35.3</td>
<td>38.0</td>
<td>43.0</td>
<td></td>
</tr>
<tr>
<td>SCC40% WG</td>
<td>24.9</td>
<td>28.0</td>
<td>30.0</td>
<td>33.0</td>
<td>35.0</td>
<td></td>
</tr>
<tr>
<td>SCC0% WG</td>
<td>31.5</td>
<td>35.8</td>
<td>40.0</td>
<td>42.9</td>
<td>47.7</td>
<td></td>
</tr>
<tr>
<td>SCC10% WG</td>
<td>30.0</td>
<td>36.6</td>
<td>40.3</td>
<td>43.0</td>
<td>48.0</td>
<td></td>
</tr>
<tr>
<td>SCC20% WG</td>
<td>28.9</td>
<td>33.0</td>
<td>36.0</td>
<td>39.0</td>
<td>45.0</td>
<td></td>
</tr>
<tr>
<td>SCC30% WG</td>
<td>29.0</td>
<td>32.8</td>
<td>36.0</td>
<td>38.3</td>
<td>43.2</td>
<td></td>
</tr>
<tr>
<td>SCC40% WG</td>
<td>27.0</td>
<td>31.0</td>
<td>34.0</td>
<td>35.9</td>
<td>38.0</td>
<td></td>
</tr>
</tbody>
</table>

For 30% WG, -12.42%, -9.34%, -10.00%, -7.14% and +4.9%, respectively. Thus, waste glass can be added successfully to the matrix with 10% MS up to 40% with improvement in compressive strength at 56 days. When the result is compared with 10% MS without waste glass, the disparity in percentage at 7, 14, 21, 28 and 56 days are as expressed below; -7.88%, -4.76%, -5.78%, -6% and -4.16% for 10% WG; -13.04%, -10.00, -9.64%, -8.40% and -9.58% for 20% WG; -18.48%, -12.86%, -13.70%, -14.00% and -18.64% for 30% WG; -21.47%, -21.43%, -22.91%, -22.00% and -28.44% for 40% WG, respectively. Hence, there are significant downturns in compressive strength when compared with the 10% MS 0% WG. These reductions were however less than those of Mix A.

For Mix C, the disparities in compressive strength are expressed in percentage of CM at 7, 14, 21, 28 and 56 days as follows; -4.55%, +1.65%, +2.5%, +4.76%, and +16.55% for 10% WG; -9.09%, -1.65%, 0.00%, +2.14% and +11.19% for 20% WG; -14.48%, -12.86%, -13.70%, -14.00% and -18.63% for 30% WG; -18.92%, -21.52%, -22.73%, -23.62% and -28.44% for 40% WG. Thus, the compressive strength fell for all substitution of river sand with WG at all ages.
These reductions were in line with the findings of Vikraman and Nithya (2015) and Sameer et al. (2016), who observed reduction in compressive strength as WG content soared. However, these contradicted the findings of Kumar et al. (2016) and Abin et al. (2017) that the Compressive strength was better with increase in substitution of fines with quarry dust (up to 30%) and iron filings (up to 40%), respectively.

5. Conclusions and Recommendations

From the results of the experiments conducted on the suitability of waste glass as partial replacement for fines in self-compacting concrete matrix blended with micro silica, the following conclusions were made:

1. The amount of Silica, aluminum oxide and ferric oxide content was beyond 70% for MS and WG. Thus, the materials can be handled as pozzolanic material founded on ASTM C618 standard (ASTM, 1994a). The materials were chiefly composed of SiO2. Micro silica and waste glass had 91.2% and 72.2% of SiO2 content, respectively.

2. Micro silica is reactive with Ca(OH)2 fixed mg of 1267.1 mg.

3. At 5%, 10% and 15% replacement of cement with MS without inclusion of WG, the rheological characteristics were affected. The slump flow downturned with increased replacement, T900mm and V-funnel flow time enlarged with replacement, while L-box value decreased. The reverse was the case when WG were added. This effect was most noticeable at 5% MS 40% WG replacement. This is due to the high water absorption capacity of MS and lower water absorption capacity of WG compared to River sand as evident in water absorption test. All the various mix passed the EFNARC (2002) requirements for fresh properties except 15% MS (mix id SCC0 and SCC10% WG), with L-box values (0.76 and 0.79) less than the recommended EFNARC (2002) standard.

4. For MS without WG, the compressive strength of MIX B 10% MS (55.3 MPa) was the highest when compared with the control (42.9MPa), MIX A 5% MS (51.3 MPa) and MIX C 15% MS (53.1 MPa) at 56 days curing. MIX B 10% MS without WG had 28.9% increase in compressive strength when compared with the control mix.

5. When WG (10-40 % at 10% interval) was introduced into the matrix, the compressive strengths of MIX B 10%MS (53, 50, 48 and 45 MPa, respectively) were higher than the control (42.9 MPa). However, the compressive strengths for MIX A 5% MS (52, 48.7, and 43 MPa) and MIX C 15% MS (50, 47.7 and 43.2 MPa) were higher than the control except for 40% WG (35 MPa for MIX A 5% MS and 38 MPa for MIX C 15% MS) at 56 days. Thus, MIX B 10% MS was the best mix.

6. In terms of compressive strength, MIX B 10% MS (with and without WG) possessed the best rheological properties.

Further work should be conducted in the areas of durability and effect of elevated temperature on the compressive strength of the matrix.

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Assessing Human Factors at the Design Stage of Upstream Oil and Gas Projects: A Risk Assessment Methodology

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Abstract: Examination of major accident data in the upstream oil and gas sector reveals the majority of cases. The initiating cause of catastrophic accidents is attributable to human error. Due to the devastating effects of these failures on people, property and the environment, current engineering design practices in the sector need to be revised in order to incorporate human factors in design considerations. A standardised risk assessment approach could be modified to make it specific to address human factors at the design stage in the oil and gas industry. This paper aims to utilise the approach to develop a proposed methodology to assess human factors, thus making the engineering design safer. The methodology constitutes 9 steps to evaluate human factors and to assess the risk posed by human error in the oil and gas sector. A case study of installing a pressure transmitter on a flowline of the oil and gas platforms was used, and an activity nonetheless which if performed incorrectly can result in a loss of containment of hydrocarbon gas. Utilising the proposed methodology, the activity’s constituent tasks are diagnosed and each task would now be subject to analysis by showing the consequences of human error. The proposed methodology can be adopted by industry to address human error in the early design phase, and would thus prevent major accidents in the upstream oil and gas sector.

Keywords: Human Factors Risk, Design Stage, Oil and Gas Projects

1. Introduction

Examination of major accidents which have occurred in the oil and gas industry often leads to the inevitable question - why did the accident occur? What are the root causes which led to the catastrophic event? Systematic review of data collected from incidents often reveals one of two categories of causation – inherent equipment failure or human error. Industry has advanced to ensure that machinery, technology and equipment are now made inherently safer (Lee, 1998). As a result, the frequency of technological failures has diminished, allowing the role of human error to become much more apparent (Gordon et al., 1996). In fact, human factors are attributed to be the root cause of 80-90% of accidents (Reason, 1997). Despite being the cause of between 80-90% of accidents, human factors are not given as much consideration as equipment failure as a root cause of accidents in the oil and gas industry.

Given that there is now a proven direct correlation between human factors and accident causation, there is a need to address human factors in these industry sectors. To date, treatment of human factors has been reactive, focusing on the management of human error rather than its prevention. In fact, there is increasing emphasis in industry to proactively address human factors issues (Embrey and Zaed, 2007).

To adopt a proactive approach, the phase at which human factors is considered is critical. The earliest phase possible to incorporate human factors in a project is the design phase. This paper will examine current engineering design practices, and explore how they can be modified and improved to include human factors. It is acknowledged that at present, there is some treatment of human factors in design. However, the application remains inconsistent and there are still barriers to implementation (Robb and Miller, 2012). This paper attempts to address these issues by developing a methodology for evaluating human factors and risk issues in design in oil and gas projects. This ability to assess the magnitude of the risks posed by human errors would enable the design team to produce a design which lowers the risk to as low as reasonably practicable thus making the design safe for use.

2. Human Factors Engineering

The International Association of Oil and Gas Producers (OGP) defines Human Factors Engineering (HFE) as a discipline which focuses on the application of human factors knowledge to the design and construction of socio-technical systems. The objective is to ensure systems are designed in a way that optimises the human contribution to production and minimises potential for design-induced risks to health, personal or process safety or environmental performance (OGP, 2011). Human factors engineering has become more crucial as evidenced during the operations phase of oil and gas projects. Operators are tasked with
troubleshooting problems in the process in order to identify deviations and suitably fix these problems coherently with the severity of expected consequences (Leva et al., 2015). This underscores the need for adequate consideration at the design stage, not only for designing for proper operability and maintainability but being able to quantify the risks associated with the postulated hazardous scenarios. By being able to quantify these risks, the appropriate safeguards and mitigation can be engineered into the design thus attenuating or eliminating the hazard.

The typical engineering design process for an oil and gas project is illustrated in Figure 1. As demonstrated in the process, the studies which are performed and the deliverables which are produced are discipline specific and discipline centric (Baron, 2015). There is little to no treatment of human factors – even under the safety function. At present, some or all of these activities are applied to engineering projects, however, they are not applied consistently. Additionally, treatment of the process is often qualitative with no quantification of risks posed by tasks performed such as those enumerated in the Tasks Requirements Analysis (TRA). Failure to risk assess tasks could potentially result in improper allocation of resources to areas which actually require them and lead to poor or under-design.

3. Assessing Human Factor Issues in Engineering Design

There have been attempts in the past to identify human error, typically termed human error identification (HEI) using task analysis (Shorrock and Kirwan, 2002). These include SHERPA (Embrey, 1986), GEMS (Reason, 1990), CREAM (Hollnagel, 1998) and HEIST (Kirwan, 1994). However, Johnson (1999) contends that these approaches have had little impact upon many industries. Additionally, Shorrock and Kirwan (2002) acknowledge that the transfer of the technology to the design and operation of safety-critical interactive systems has encountered serious problems.

Figure 1 illustrates the engineering deliverables produced during the design phase. These typically encompass studies and drawings performed for the process, mechanical, electrical and instrumentation, civil/structural, piping, and safety disciplines. It is interesting to note that there is a lack of human factors studies and/or considerations in the process.

A standardised risk assessment approach as advocated in literature (Moore, 2003) could be modified to make it specific to address human factors at the design stage in the oil and gas industry. The approach is utilised for developing the proposed methodology as it is common in the oil and gas sector to employ risk assessment in hazard evaluation. Figure 2 is a flow chart depicting the proposed steps to evaluate human factors in engineering design and to assess the risk posed by human error. The steps involved in the process are:

1. Identification of activities to be carried out,
2. Identification of tasks in the activities,
3. Opportunities for human error,
4. Performance Influencing Factors (PIFs),
5. Determination of consequences of PIFs,
6. Human error risk ranking,
7. Controls and Mitigation measures,
8. Human error risk ranking after mitigation, and
9. Recommendations to be incorporated in design.
3.1 Identification of Activities
Generally, for oil and gas projects, activities to be performed can be divided into one of the top level categories outlined as follows:
- Management of Platform Safety
- Maintenance of Platform
- Drilling operations
- Monitor and Control Platform status
- Planning and Administration
- Domestic Activity

From these top-level categories, various activities are carried out. These activities comprise various tasks which are performed by operations and maintenance personnel either employed or contracted by the organisation.

3.2 Identification of Tasks in Activities
Each activity can now be divided into its constituent tasks. Essentially, task identification entails where humans interact with any process identified in the broad activities. Most of this information can be garnered from operating procedures and maintenance schedules. For greenfield projects, in the absence of existing procedures and schedules to consult, the tasks can be obtained via brainstorming sessions from subject matter experts and experienced professionals who would have knowledge in these areas.

3.3 Performance Influencing Factors
Once the tasks have been identified, using a prescribed list of Performance Influencing Factors (PIFs) can help to identify the factors which can influence behaviour but more specifically, influence behaviour in a negative way. The United Kingdom Health and Safety Executive (UK HSE) defines PIFs as the characteristics of the job, the individual and the organisation that influence human performance. Optimising PIFs will reduce the likelihood of all types of human failure. PIFs generally include:
- **Individual factors** - Low skill and competence levels. Tired, bored or disheartened staff. Individual medical problems.
- **Organisational factors** - Poor work planning, leading to high work pressure. Management based upon one-way communications. Deficient co-ordination and responsibilities. Poor health and safety culture.

These failures as described by the UK HSE can be unintentional i.e. a skill-based error or mistake or they may be intentional which is defined as a violation. Human errors

![Figure 2. Human Failure as Outlined by the UK HSE](image)

3.4 Opportunities for Human Error
The opportunities for human error include the areas where an operator can introduce an error leading to an active failure.

3.5 Determination of Consequences of PIFs
Correctly identifying PIFs helps to determine the activities which could directly cause a major injury, death or loss of hydrocarbons to the environment, have significant business impact (including production outages) or could contribute to significantly increasing the frequency or consequence potential of the incidents. All PIFs do not have major consequences though. For this reason, the consequences will be ranked from those having minor to major impacts.

3.6 Human Error Risk Ranking
In order to adequately assess the consequences caused by human error, a semi-quantitative approach which relies on historical data, as well as the expertise of oil and gas professionals has been developed. A matrix such as this one shown in Figure 3 is widely used in the industry and can be adapted or amended to suit the facility.

![Figure 3. Risk Rank Matrix](image)
The consequences of human error are rated from low to high and an appropriate likelihood of this impact assigned. The risk rank is then determined as a product of the consequence and likelihood and can range from low to critical. If risks are deemed low to medium, the task can proceed with caution and the relevant mitigatory measures implemented. If risks are deemed high to critical, additional recommendations are necessary including reviewing and revising the design to lower the risk to an acceptable level.

### 3.7 Controls and Mitigation Measures

The controls and mitigation measures will include measures or design features which will prevent the top event from occurring or attenuate the consequences of the event if the top event does occur. In Human Factors Engineering, most mitigation measures tend to be training or procedural based. However, it is hoped that by employing this proposed risk assessment technique at the design stage, the controls and mitigation measures to be proposed would be design-based or modifications made to the design in order to attenuate the risk due to human error.

### 3.8 Human Error Risk Ranking after Mitigation

Just as the human error risk is ranked pre-mitigation, the risk needs to be assessed post mitigation in order to determine if the mitigation measures proposed are deemed to be adequate or tolerable. This step involves the same process as that involved in ranking the risk pre-mitigation.

### 3.9 Recommendations Incorporated in Design

Figure 4 shows a flowchart of steps to evaluate human factors in engineering design. If the human error risk is deemed to be too high or critical, the design must be modified to prevent the human error from occurring. Recommendations are made at this stage to revise the design to make it safer for use.

### 4. Application of the Risk Assessment Methodology – A Case Study

It is proposed to install a pressure transmitter on a flowline on an existing ½” bleed point connection. The tasks involved in the job are outlined in Figure 5. The proposed methodology is applied to the tasks. The outcome is illustrated in Table 1. From the table, the broad activity is identified as the installation of a pressure transmitter – a standard activity on oil and gas platforms but an activity nonetheless which if performed incorrectly can result in a loss of containment of hydrocarbon gas. Utilising the proposed methodology, the activity’s constituent tasks are outlined as shown in Figure 3 but each task is now subject to analysis by showing the consequences of human error. The credible human error is identified based on the UK HSE’s breakdown of human failure (HSE, 2018).

The human error may take the form of slips of actions, lapses of memory or mistakes. Using these Performing Influence Factors (PIFs) helps to postulate scenarios which can result in deviations from normal operating procedures or accidents. It is then easier to evaluate the consequences of the accidents. The consequences need to be risk ranked in order to determine the severity and so provide an indication as to what controls are necessary to prevent them from occurring or mitigate the consequence should the accident occur.

When this methodology is utilised, there are several outcomes which are noted. The methodology helps the process to systematically identify the human error, the consequence and the risk associated with the consequence. This information can then be used to influence the design at an earlier stage so that the human error can be taken into account. By rectifying the design, the post mitigated risk is then seen to be lowered to an acceptable level making the design safe for use and enabling the activity to be carried out safely.
### Table 1. Human Factors Analysis of Case Studies

<table>
<thead>
<tr>
<th>Activity</th>
<th>Task</th>
<th>Performance Influencing Factor</th>
<th>Opportunity for Human Error</th>
<th>Consequence</th>
<th>Pre Likelihood</th>
<th>Pre Impact</th>
<th>Pre Risk Rank</th>
<th>Mitigation</th>
<th>Post Likelihood</th>
<th>Post Impact</th>
<th>Post Risk Rank</th>
<th>Recommendation</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. Install pressure transmitter on flowline</td>
<td>1. Purge and isolate flowline</td>
<td>Slip - Operation omitted</td>
<td>Operator fails to isolate flowline</td>
<td>On removal, loss of containment - size of loss dependent on process area.</td>
<td>M</td>
<td>M</td>
<td>M</td>
<td>Competency, procedural control. SSOW.</td>
<td>L</td>
<td>M</td>
<td>L</td>
<td>Ensure that DCS design is configured so that pressure indication can be read upstream and downstream of installation site so that continuous pressure monitoring is achieved</td>
</tr>
<tr>
<td>2. Remove bolt from 1/2&quot; bleed point</td>
<td>2. Wrong operation on right object</td>
<td>Bolt or spanner is dropped onto equipment / live line below during removal or handling</td>
<td>Direct injury to personnel or loss of containment / explosion (or both).</td>
<td>M</td>
<td>H</td>
<td>H</td>
<td>Lifting procedures. Controlled by ISSW. Permit checked by supervisor on completion.</td>
<td>L</td>
<td>M</td>
<td>L</td>
<td>All lines, instruments and connection point should be properly labelled and labelling reconciled and correspond to As-built drawings so that there is no ambiguity with regards to where works should be performed. This is informs the need to depressurise / purge lines in the vicinity of works to be carried out</td>
<td></td>
</tr>
<tr>
<td>3. Install pressure transmitter</td>
<td>3. Wrong operation on right object</td>
<td>Bolt or spanner is dropped onto equipment / live line below during removal or handling</td>
<td>Direct injury to personnel or loss of containment / explosion (or both).</td>
<td>M</td>
<td>H</td>
<td>H</td>
<td>Lifting procedures. Controlled by ISSW. Permit checked by supervisor on completion.</td>
<td>L</td>
<td>M</td>
<td>L</td>
<td>See Recommendations 2 and 3</td>
<td></td>
</tr>
<tr>
<td>4. Commission instrument</td>
<td>4. Loop - Check omitted</td>
<td>Operator fails to carry out line walk, or incomplete / non-compliant line walk</td>
<td>Undetected issue with PT, may escalate to major accident hazard</td>
<td>H</td>
<td>M</td>
<td>H</td>
<td>Controlled by ISSW. Permit checked by supervisor on completion. LOLR register where relevant.</td>
<td>M</td>
<td>M</td>
<td>M</td>
<td>Consider equipment redundancy so that if equipment is faulty, the back up will function as expected during operations</td>
<td></td>
</tr>
</tbody>
</table>
This proposed methodology makes allowances for recommendations of post-mitigation. Although procedural controls and supervision may historically be deemed sufficient to control risks associated with certain activities, the objective of any operation is to ensure that in its operation, the risk is sufficiently lowered to as low as reasonably practicable. As seen from the case study, although lifting procedures, permit checks and safe systems of work are deemed sufficient to remove the bolt from the bleed point as evidenced by the acceptable low risk achieved post mitigation, this methodology still makes allowances for design modification, if necessary, following the completion of a dropped objects study report which would highlight any dropped object risk which may affect the pipeline on which work is being performed or other pipelines in the vicinity of the work. This shows that this methodology helps to properly evaluate human error and its consequences as well as influence the design in a proactive way which would streamline the design process, make it more efficient, save on cost and ultimately make it safe.

6. Conclusion

The proposed risk assessment methodology was modified to include human factors. It was developed using a modified approach proposed by Moore (2003). The methodology attempts to delineate human error and the performance influencing factors, as well as incorporate the post-mitigation risk rank.

Utilising this methodology, the human errors which occur during various activities performed by operators/other platform personnel, could be identified. With identification of human errors, the consequences as a result of the failures can be enumerated and appropriately risk ranked. The risk rank also informs the magnitude and scope of the control or mitigation measures necessary to prevent or attenuate the impact. More importantly though, the procedure allows for making recommendations which ultimately produces more robust, fail safe designs with little opportunity for human error.

This procedure also allows for early identification of human error so that the design considers various modes of human error and design to prevent these errors from taking place. Traditionally, the user interface is studied further along in the design process which sometimes prevents adequate treatment of human error in the design phase. This means that by the time the human error is identified, the design has gone through the design stage and is operational. Designs must then be retrofitted, modified or upgraded to take into account the human error which can be difficult given that space is a limited commodity on offshore oil and gas platforms and redesigning is often costly. By analysing the human error early in the design phase, these issues can be addressed. Therefore, the controls used to prevent or mitigate human error are not limited to procedural or administrative controls such as ‘Safe Systems of Work’ (SSOW) or permits as seen in Table 1, but can also include engineering design solutions.

This procedure demonstrates that human error can be assessed at the design stage of a project, thus enabling the designer to modify the design to suit the user and so eliminate/reduce the risk associated with the activity. It can also eliminate the need for retroactive design where human error was not adequately addressed. This procedure is easy to use, and can be integrated into any organisation by the use of simple spreadsheets or software.

References:


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Assessing Small Enterprises’ Maturity Levels of Knowledge, Attitudes and Practices towards Emergency Preparedness in Trinidad: A Pilot Study

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Abstract: This paper aims to examine small enterprises’ knowledge, attitudes and practices (KAP) towards emergency preparedness in Trinidad and Tobago (T&T). A pilot study comprising survey and interviews was used, and a group of three small enterprises were invited. It was found that lack of legislations, limited financial resources and little institutional guidance were amongst the main challenges for small enterprises in engaging emergency preparedness operations (EPO). Based on the pilot findings, a 5-level Generic Emergency Management Assessment (GEMA) approach was developed with implementation guide. The approach would serve as a guidance tool for small enterprises to 1) assess their current maturity level, 2) implement programme requisites to bridge gaps, and 3) move from one maturity level to the next. Future work would expand the testing of the approach with wider enterprise base. Its applicability to improve emergency preparedness in medium and large organisations would be explored.

Keywords: Emergency preparedness; small enterprises; maturity levels, EPO, KAP, GEMA

1. Introduction

The Caribbean Region is made up of small island developing states (SIDS) that are vulnerable to natural disasters such as floods, landslides, hurricanes, earthquakes and intermittent volcanic eruptions. According to Collymore (2011), disaster events would cost the region approximately US$3 billion on an almost annual basis. Being one of SIDS in the Caribbean region, the Republic of Trinidad and Tobago (T&T) has been susceptible to natural disaster events which have significant negative consequences more than that previously realised by the general public (Carby, 2011).

In T&T, small enterprises in the energy sector have to attain Safe-to-Work (STOW-TT) certification (STOW, 2019). One of the elements required to meet STOW-TT minimum requirements is the implementation of Crisis and Emergency Management. Recent studies suggested that measures to assist emergency preparedness operations (EPO) would include the passing of disaster preparedness legislation, dissemination of relevant information and provision of technical assistance, incentives for adoption of preparedness initiatives (Collymore, 2011) and demonstration of the net financial benefits (Mohan et al., 2013). However, majority of these enterprises do not have the resources or knowledge to undertake risk assessments and develop emergency preparedness plans in T&T (Collier, 2015).

EPO involve persons acting systematically to minimise loss of life, property damage and environmental impact from an emergency event. Incorporating the elements of NFPA 1600 standard (NFPA, 2013, 2019) and a determination of maturity levels as advocated by Jones (2003). There has been a need for these enterprises to improve their EPO performance. This paper aims to assess the maturity levels of enterprises’ knowledge, attitudes and practices (KAP) towards EPO in T&T. A Genetic Emergency Management Assessment (GEMA) approach was developed, with a set of implementation guideline and performance metrics.

2. Emergency Preparedness and NFPA 1600 Standard

Emergency preparedness involves implementation of the processes of planning, training and drills in combination with the acquiring of necessary equipment and resources to enable a desired emergency response to be mounted (Perry and Lindell, 2003). Considerations should include natural, man-made, and technology-related threats. Moreover, preparedness is a dynamic process which includes making the plan known through training and ensuring that resources and response equipment are available. Emergency preparedness NFPA 1600 (Standard on Disaster/Emergency Management and Business Continuity/Continuity of Operations Programmes) is a standard published by the National Fire Protection Association. An all-hazard approach as outlined in NFPA 1600 can be used to guide emergency preparedness efforts (Adni et al., 2012; NFPA, 2019).

According to the NFPA 1600 standard (NFPA, 2013, 2019), six (6) elements of an emergency preparedness programme were suggested for small businesses. These
include:
1) Programme management – This shows the commitment of the leadership of the organisations to the emergency management programme.
2) Planning – This includes the development of emergency plans based on the undertaking of risk assessments and business impact analyses (BIA).
3) Implementation – This identifies roles and responsibilities of persons within the organisation and agencies external to the organisation.
4) Training and Education – This ensures that training is provided to employees appropriate to their level of involvement in the programme.
5) Exercises and Tests – This requires organisations to practice their plan and preparedness through the implementation of drills and exercises.
6) Programme Maintenance and Improvement – This includes regular review of the emergency programme, implementation of lessons learned, and continuous improvement of programme.

3. Maturity Levels of Emergency Preparedness
Policies and legislations should be made to support a nationally institutionalised framework. According to the Office of Disaster Preparedness and Management of T&T (ODPM, 2019), a comprehensive disaster management plan could outline the roles and responsibilities of stakeholders. It is essential to facilitate the collation and sharing of information on best practices and lessons learnt from EPO between stakeholders. Nevertheless, most small enterprises have limited resources to develop EPO (IDB, 2010), and a significant hindrance to the growth of small enterprises was the access to financing (Joefield, 2016).

Jones (2003) advocated the concepts of the Capability Maturity Model (CMM), and developed a framework for assessing the emergency management system in organisations. The CMM was initially employed in the information technology (IT) industry to improve the process of software development and assess the improvement capability (Yeh et al., 2017). According to Janse (2018), the CMM consists of five (5) levels which reflect progressively improving processes. In relation to emergency preparedness these levels are described briefly below (Raeburn-Marcano, 2019):

- Level 1 – Initial (ad hoc): This level reflects emergency preparedness operations (EPO) in the early phase of development where there is little or no documentation and processes are mainly reactive.
- Level 2 – Repeatable: This is the stage during which goals are set, response requirements are determined and roles are defined.
- Level 3 – Defined: This represents fully developed EPO which is integrated into the organisation’s processes and activities.
- Level 4 – Managed: This reflects the stage at which processes are fully established and improvement opportunities are identified for optimisation of processes.
- Level 5 – Optimising: This is the stage at which processes within the EPO are proactively improved even when new equipment or changes to operations occur(s).

An organisation could determine its current or baseline state of emergency preparedness, and then set goals and objectives to ascend to the higher levels (Paulk, 2009). For facilitating the CMM adoption, key EPO process areas are to be identified, and a three-pronged sequence of preparedness, response and learning is to be determined. Moreover, maturity could be assessed with reference to three (3) dimensions, namely knowledge, attitude and practice. As contended by Mohanty et al. (2006), Knowledge is the fact or condition of knowing something with a considerable degree of familiarity through experience, association or contact. Attitude is a mental position representing a willingness to carry out varying tasks, whereas Practice is the actual undertaking or doing of tasks or activities (Andersen and Jessen, 2003). An organisation is said to be of mature state when its objectives are fulfilled. Many organisations are continually developing or improving, and it would therefore be better to focus on the levels of maturity attained. Organisations could monitor development of a process from an initial level to progressively improving stages of maturity (Andersen and Jessen, 2003).

4. A Pilot KAP Study in T&T: Methods and Procedures
A pilot Knowledge-Attitude-Practice (KAP) survey was undertaken during the months of March and April 2019 in T&T. It aimed to acquire both qualitative and quantitative information to examine the current challenges and problems experienced by small enterprises in managing emergencies, and to develop an approach/guide to track their EPO practices (MdM, 2012; Raeburn-Marcano, 2019). A non-probability purposive sampling technique was adopted (Naser and Saleem, 2018), and three (3) small private enterprises operating at Port-of-Spain, the capital city of T&T, were invited. A total of thirty-eight (38) individuals representing the total number of employees hired by these three enterprises participated in the study. These companies (represented by ‘A’, ‘B’ and ‘C’) were drawn from respective sectors of manufacturing/processing, healthcare, oil and gas.

The study consisted of 1) a questionnaire survey with close-ended questions, and 2) a series of structured interviews, with the safety managers, staff and/or other delegated representatives of participating small enterprises. A set of self-administered questionnaires was developed based on previous knowledge, attitude and practice (KAP)
studies undertaken involving healthcare workers in Lagos (Adenekan et al., 2017), Ethiopia (Habte et al., 2018) and Delhi (Yadav et al., 2016). The instrument comprised 28 closed-ended questions in four (4) sections. These are: 1) Demographic data, 2) Knowledge check, 3) Attitude check, and 4) Practice check, pertaining to assessing awareness and maturity levels of EPO in organisations. For conducting the interviews, an instrument comprising 14 closed- and open-ended questions, was used to acquire the management’s views on the implementation of emergency plan, the risk assessments and drill exercises, and various EPO challenges being faced by small enterprises.

In determining the ‘knowledge’ score, one score point was assigned to each answered question, whereas no score would be given to those unattempted questions and/or questions with incorrect answers. The maximum score was eleven (11). Participants with a knowledge score of 9 or higher were considered “good” performers (Naser and Saleem, 2018). For the ‘attitude’ score, a maximum score was 32. Respondents obtaining a score of 26 or more were of positive attitude towards EPO. For the ‘practice’ score, the maximum was twelve (12), and a score of ten (10) or more was considered as “good”. Moreover, a Chi-squared test was used to examine the relationship of knowledge, attitude and practice between the study variables (Raeburn-Marcano, 2019).

5. Findings and Analysis
5.1 Response Rate and Demographics of Respondents

In total, thirty (30) completed questionnaires out of 38 were received, yielding a response rate of 79%. The reliability of the data was checked, and the result obtained was a Cronbach's Alpha value of 0.568. Table 1 depicts the breakdown of respondents by demographics.

<table>
<thead>
<tr>
<th>Variable</th>
<th>n</th>
<th>%</th>
</tr>
</thead>
<tbody>
<tr>
<td>Age in years</td>
<td></td>
<td></td>
</tr>
<tr>
<td>≤ 30</td>
<td>6</td>
<td>20.0</td>
</tr>
<tr>
<td>&gt;30</td>
<td>24</td>
<td>80.0</td>
</tr>
<tr>
<td>Years at company</td>
<td></td>
<td></td>
</tr>
<tr>
<td>≤ 5</td>
<td>7</td>
<td>23.3</td>
</tr>
<tr>
<td>&gt;5</td>
<td>23</td>
<td>76.7</td>
</tr>
<tr>
<td>Previous work exp.</td>
<td></td>
<td></td>
</tr>
<tr>
<td>1-5</td>
<td>13</td>
<td>43.4</td>
</tr>
<tr>
<td>&gt;6</td>
<td>9</td>
<td>23.3</td>
</tr>
<tr>
<td>Gender</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Male</td>
<td>14</td>
<td>46.7</td>
</tr>
<tr>
<td>Female</td>
<td>16</td>
<td>53.3</td>
</tr>
<tr>
<td>Position in Organisation</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Management</td>
<td>7</td>
<td>23.3</td>
</tr>
<tr>
<td>Administrative staff</td>
<td>7</td>
<td>23.3</td>
</tr>
<tr>
<td>Support Services</td>
<td>12</td>
<td>40.0</td>
</tr>
<tr>
<td>Education level</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Primary</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>Secondary</td>
<td>13</td>
<td>43.3</td>
</tr>
<tr>
<td>Tertiary</td>
<td>17</td>
<td>56.7</td>
</tr>
</tbody>
</table>

Eighty percent (80%) of respondents were over thirty years of age and 76.7% had worked at their respective present organisations for over five years. Most (73.3%) had previously worked at similarly small organisations prior to their respective current employer. Just over fifty-three percent (53.3%) of participants were female. Moreover, all respondents had attained at minimum secondary school education level with 56.7% of them having attained tertiary level education. With respect to their positions, management personnel accounted for 23.3%, administrative staff represented 23.3%, and the rest were support services staff.

5.2 The KAP Findings on Emergency Preparedness

A summary of the KAP findings is depicted in Table 2, and the analysis was based on the eight (8) preparedness indicators advocated by Dahlerman and D'Souza (1997). The results indicate that the overall level of preparedness among the three companies was 52.5%, where Company C has a level of 87.5%, followed by Company A of a 50% level and the Company B of a 20% level. The average emergency preparedness level between Companies A and B was thirty-five percent (35%) (if the result from Company C was excluded). This appeared comparable to the findings from other recent studies (like, Murray and Watson (2019)) which indicated about 40-60% positive responses from large organisations on disaster/emergency preparedness. It reflected a lower level of emergency preparedness in small private-sector enterprises as compared with that in larger organisations.

<table>
<thead>
<tr>
<th>Actions</th>
<th>Company A</th>
<th>Company B</th>
<th>Company C</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. Talk with employees</td>
<td>Yes</td>
<td>Partial</td>
<td>Yes</td>
</tr>
<tr>
<td>2. Store food or water</td>
<td>Yes</td>
<td>Yes</td>
<td>Yes</td>
</tr>
<tr>
<td>3. Store battery-operated devices</td>
<td>Partial</td>
<td>Partial</td>
<td>Partial</td>
</tr>
<tr>
<td>4. Learn first aid</td>
<td>Yes</td>
<td>No</td>
<td>Yes</td>
</tr>
<tr>
<td>5. Obtain first aid kit or extra medical supplies</td>
<td>Yes</td>
<td>Yes</td>
<td>Yes</td>
</tr>
<tr>
<td>6. Documented emergency plan</td>
<td>No</td>
<td>No</td>
<td>Yes</td>
</tr>
<tr>
<td>7. Conduct drills or exercises</td>
<td>Limited, desktop drills</td>
<td>No</td>
<td>Yes</td>
</tr>
<tr>
<td>8. Involved in training preparedness or response training programmes</td>
<td>Partial</td>
<td>Limited</td>
<td>Yes</td>
</tr>
</tbody>
</table>

Moreover, the maturity level as collated from managers’ and employees’ views on KAP towards emergency preparedness was consistent with the maturity level of EPO in respective organisation. Company C in the...
oil and gas sector has a significantly higher level of preparedness than that of the other two participating companies in manufacturing/processing and healthcare sectors in T&T. Employees from Company C also exhibited a greater level of knowledge, more positive attitude and practice levels than those from Companies A and B. The data and views acquired from the pilot study was to set the baseline assessment level to which future assessments could be referenced. The analysis of data would indicate the maturity levels, with respect to knowledge, attitude and practices.

Furthermore, several gaps have been identified from the analysis, addressing the need for these enterprises to commit initiative and resources towards EPO. These included:
- A documented emergency preparedness operations plan,
- Formal system of risk assessment for emergency events,
- Structured training and development programmes,
- Continuous programme of drills and exercises,
- Regular discussions and meetings on emergency preparedness, and
- Provision of adequate resource personnel, equipment and supplies to meet needs identified in risk assessment.

6. A Proposed EPO Initiative for Small Enterprises

6.1 A Generic Emergency Management Assessment Approach

An attempt was made to identify the processes of EPO in small businesses and define the maturity levels against which these processes should be assessed. A proposed Generic Emergency Management Assessment (GEMA) approach was developed, incorporating the elements of NFPA 1600 standard (NFPA, 2019) and the structure of maturity levels as advocated by Jones (2003). There are five (5) maturity levels for the GEMA approach, including:
- Level 0: Default/Ad-hoc,
- Level 1: Functional and Repeatable,
- Level 2: Focused and Defined,
- Level 3: Managed, and
- Level 4: Optimising.

Level 0 reflects the default or ad-hoc position of not having regulatory or process requirements in place. On this basis, the Level 1 heading of “Functional and Repeatable” suggested by Jones (2003) was adopted. At Level 1, it is expected that an organisation has an emergency plan in place which is used to provide a specific response to incidents likely to occur.

At Level 2, it is expected that goals and targets are set, and considerations of capability are performance-based. At this level, the EPO is fully defined, and the focus is to ensure integration into the organisations’ processes and activities. The level heading can be termed “Focused and Defined” (Jones, 2003). At Level 3, this can be termed “Managed” and reflects the stage at which processes are fully established and improvement opportunities are identified for optimisation of processes. Lastly, the heading for Level 4 can be termed “Optimising”, as at this stage processes within the EPO are proactively improved even when new equipment or changes to operations occur(s). Adoption of the GEMA approach would support business strategy and facilitate the planning, development and implementation of tactical, project and operational objectives towards emergency preparedness.

6.2 Alignment of EPO Processes with Maturity Levels

The pilot KAP study findings were used to align processes with maturity levels in the GEMA approach. Table 3 shows the alignment of EPO processes and the assessment of the maturity level of respective processes, and by extension, the maturity level of the three (3) small enterprises under study.

<table>
<thead>
<tr>
<th>Process</th>
<th>Maturity Level 1</th>
<th>Maturity Level 2</th>
<th>Maturity Level 3</th>
<th>Maturity Level 4</th>
</tr>
</thead>
<tbody>
<tr>
<td>Programme</td>
<td>Managers with designated responsibility for the EM programme. Programme meets</td>
<td>EPO is fully developed and being integrated into company processes and activities.</td>
<td>EPO is integrated into company processes. Team meetings discuss incidents.</td>
<td>Management of Change procedure ensures company automatically and dynamically adjusts to changes.</td>
</tr>
<tr>
<td>Management</td>
<td>regulatory requirements. Records are managed.</td>
<td>Team meetings used to discuss incidents.</td>
<td>Management of Change procedure prompts review of plan as required.</td>
<td></td>
</tr>
<tr>
<td>Planning</td>
<td>Company has emergency plan based on hazard outcomes from HSE risk assessment.</td>
<td>Hazard identification and risk assessment undertaken. Performance objectives</td>
<td>Performance objectives integrated into company objectives and measured.</td>
<td></td>
</tr>
<tr>
<td>Implementation</td>
<td>Roles and responsibilities are identified in the plan. Emergency procedures are</td>
<td>Procedures to include damage assessments and resource needs assessment.</td>
<td>Management of resources integrated into company Human Resources and Purchasing</td>
<td>Management of change (MOC) procedure prompts review of roles and resources as</td>
</tr>
<tr>
<td></td>
<td>developed and emergency supplies are available.</td>
<td>Communications plan for on-the-road employees.</td>
<td>processes.</td>
<td>required.</td>
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</table>

Table 3. Alignment of EPO Processes to Maturity Levels in Proposed GEMA Approach
Six (6) EPO processes were determined, and the process activities associated with the various maturity levels could then be used as a guide. These processes were consistent with that of NFPA 1600 standard in the emergency preparedness programme for small businesses (NFPA 2013, 2019), and are listed as follows:

1) Programme Management: Leadership and management showed support for emergency preparedness activities.
2) Planning: This included formal emergency plan along with procedures, training and communication in plans.
3) Implementation: Roles and responsibilities of staff were outlined in the emergency plan.
4) Training: There was evidence of and plans for fire training, first-aid training, and recertification in training.
5) Exercises: This showed the conduct of quarterly and annual drill exercises.
6) Programme Maintenance and Improvement: This showed emergency plan and risk assessment to cater for operational changes and hazard identification.

6.3 Development of Implementation Plan
A process-flow diagram was developed as an assessment-developmental aid of the GEMA approach. Figure 1 shows the cyclic nature of the approach which stresses continuous improvement. An organisation can start at the first process activity identified (as shown in the diagram) and work its way through assessing the present status and at the same time determining the areas for continuous improvement.

Company’s mission and vision statement would reflect the desired end-state of EPO implementation. It is expected that the EPO would have enterprise-wide influence and be visibly supported by senior management. The organisational structure would be defined, and the roles and responsibility be designated to an EPO/safety manager (or a representative or delegated person). The process-flow diagram would allow for standardisation and repetition in execution to reduce cost and completion times. With the development of programmes and documentation of practices, the GEMA adoption would foster work ethics and relationships among senior management, delegated representative and staff members in developing relevant procedures and/or protocols. Moreover, key performance indicators (KPIs) could be established and then be measured, monitored and regularly reported on to senior management and other stakeholders.

6.4 Pilot Evaluation of the GEMA Approach
In the pilot KAP study, several factors affecting the adoption of EPO in small enterprises were identified. These included: 1) not legislatively required; 2) lack of institutional guidance; 3) costly to implement; 4) not a money-generating endeavour; and 5) time-consuming. Small enterprises could use the GEMA approach to assess their present maturity level and chart the EPO way forward. It was found that most of the EPO process activities were not consistently implemented in both Companies A and B, and their EPO maturity was assessed at Level 0. For Company C, most of its EPO process elements could be identified and its EPO maturity had attained Level 1. These elements included:

1) Emergency plan to be reviewed annually,
2) Training plan and matrix with provisions for recertification and refresher training,
3) Plans and procedures for the hazards identified,
4) Roles and responsibilities of staff are outlined in the plan, as well as the process for callout,
5) Identification of hazards to be based on risk assessment outcomes, and
6) Fire detection and alarm system installed onsite and internal discussions to be held after incidents.

Based on the pilot findings, the GEMA approach would serve as a guidance tool for small enterprises to develop business continuity plans and to better determine resource requirements for attaining emergency preparedness.
Company designates person with responsibility for GEMA

Company expands mission and vision statement to include EPO

Company establishes goals and objectives for EPO

Develop EPO Org Structure and Assign Roles and Responsibilities

Set KPIs/ targets for Risk Assessment Review

Identify hazards and assess risks to people, property and environment

Determine maturity level of Programme Management

Implement improvement initiatives to Planning and Implementation

Set KPIs/ targets for Plan review

Evaluate adequacy of Plans and Procedures

Determine maturity level of Planning and Implementation

Implement improvement initiatives to Training

Set KPIs/ targets for Training

Examine acceptability of Training Plan

Determine maturity level of Training

Implement improvement initiatives to Exercises

Set KPIs/ targets for Exercises

Evaluate adequacy of Exercises

Determine maturity level of Exercises

Implement improvement initiatives to Programme Maintenance and Improvement

Set KPIs/ targets for Programme improvement

Examine acceptability of Programme Maintenance and Improvement

Determine maturity level of Programme Maintenance and Improvement

Implement improvement initiatives to Programme Maintenance and Improvement

**Symbol** | **Representation**
---|---
1 | Process 1: Programme Management
2 | Process 2: Planning
3 | Process 3: Implementation
4 | Process 4: Training
5 | Process 5: Exercise
6 | Process 6: Programme Maintenance and Implementation
| | Activity associated with Process
| | Process flow
| | Process/ Information flow

**Figure 1:** Process-Flow Diagram of Implementation Plan for Proposed GEMA Approach
7. Conclusion

In T&T, most of the small enterprises have limited financial resources and lacked institutional guidance to aid in the implementation of EPO. It is however, in their best interest to implement emergency preparedness measures. This paper examined the knowledge, attitudes and practices (KAP) of selected small enterprises towards emergency preparedness. Among the most detected gaps found would be lack of:

1) A documented emergency preparedness operations plan,
2) Formal system of risk assessment for emergency events,
3) Structured training and development programme,
4) Continuous programme of drills and exercises,
5) Regular discussions and meetings on emergency preparedness, and
6) Provision of adequate resource personnel, equipment and supplies to meet needs identified in risk assessment.

The pilot study shared insights on how to assess the maturity level and provided some findings on how these enterprises engage in EPO in T&T. Incorporating the findings with desk research, the GEMA approach was developed along with a set of implementation guide. There were five (5) levels of maturity, comprising Level 0: Default/Ad-hoc, Level 1: Functional and Repeatable, Level 2: Focused and Defined, Level 3: Managed, and Level 4: Optimising. The approach aligns six processes identified to assess the maturity level of a process in a company’s EPO and by extension the maturity level of the organisation.

With the adoption of the GEMA approach, it is anticipated that small enterprises would be able to 1) assess their current maturity level, 2) implement programme requisites to bridge gaps, 3) move from one maturity level to the next, and 4) share knowledge and continuously improve and could progressively develop an affordable emergency preparedness programme, utilising existing resources and planning opportunities. Future work should expand the acquisition of empirical findings and cases with a wider population of small enterprises from various sectors. Findings of associated EPO challenges and measures of maturity levels using the GEMA approach would be compared for consistency. The applicability of the GEMA approach to medium-sized and/or large organisations could also be explored in T&T and a wider Caribbean region.

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